# ALKALI STABILIZED FLY ASH: A NEW GENERATION GEO-MATERIAL

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# CERTIFICATE

This is to certify that the thesis entitled **"ALKALI STABILIZED FLY ASH: A NEW GENERATION GEO-MATERIAL"** being submitted by **Swaraj Chowdhury** bearing Roll No. 213CE1041 towards partial fulfilment of the requirement to award the degree of Master of Technology in Geotechnical Engineering at Department of Civil Engineering, National Institute of Technology Rourkela is a record of bonafide work carried out by him under my guidance and supervision. It is further certified that the contents presented in this thesis has not been submitted elsewhere for the award of any degree or diploma.

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## ABSTRACT

In India most of thethermal power plants are dependent on coal. Out of the total power generated, 57% of the power generated is from coal-based thermal power plant. The bituminous type coal used in India, the coal ash content is in the range of 30% to 50%. The operating conditions of thermal power plantsand the quality of coal used affects the quantity of fly Ash produced. The annual production of fly Ash presently in India is more than110 million tonnes and the land occupied by ash ponds is 65000 acres and the fly ash production is likely to cross 225 million tonnes by the year 2017. Such a vast quantity of fly ash does cause challenging problems , in the form of land usage , health risks , environmental hazards and disposal . Both in disposal as well as in utilization , extreme care has to be taken forprotecting the interest of human life and environment .

Fly ash is toxic in nature , easily combustible , corrosive and reactive resulting it posses harmful effects on the environment . Fly Ash particles size varing from 0.5 to 300 micron in equivalent diameter , being light weight , have prospective to get airborne easily and contaminate the environment . If not managed suitably fly ash disposal in sea /rivers/ ponds can cause harm to aquatic life also . It can also pollute the under - ground water resources with hints of poisonous metals present in it .

Therefore proper disposal of fly ash is one of the major concerns to be dealt with; hencean advanced solution which would be effective, proficient and environmentally appropriate is required to overcome this difficulty of fly ash disposal . So , with suitable stabilization fly ash can be used as a substitute geo - material in many civil engineering construction .

For promoting the usage of fly ash as one of the leading construction material or geo - material, it is advisable to improve its engineering properties by stabilizing it with suitable stabilizer. Typically fly ash doesn’t possess strength itself; usually we used lime (CaO) and ordinary Portland cement (OPC) as additive for stabilizing fly ash. But a major issue with OPC is that its manufacturing processes are energy demanding and emit a large quantity of carbon di-oxide ($CO_2$). For example, roughly one ton of $CO_2$ is released for the manufacture of one ton of cement. Also, the raw materials available for cement

production are being over-consumed while lime (CaO) reacts very slowly with fly ash to form pozzolanic material so it will take more time to achieve desired strength. Hence, civil engineering field is always in search for new, viable sustainable fly ash stabilizer.

The present work aims at assessment of the effectiveness of adding alkalis ( i.e. NaOH , KOH and $Ca(OH)_2$ ) in stabilizing the fly ash and its appropriateness to be used as a construction material or geo - material . Fly ash used for research in this project was collected from the thermal power plant of CPP - NSPCL , Rourkela Steel Plant . For assessing the suitability of any construction material for various geotechnical engineering works its compaction properties , strength parameters and hydraulic conductivity properties are the most important properties to be tested .

In this project , an effort was made to assess the index , chemical and engineering properties of fly ash and alkali stabilized fly ash . First the physical , chemical and engineering properties of the fly ash samples were studied by conducting grain size distribution , hydrometer analysis , heavy compaction test , UCS test , falling head Permeability test and pH test of original fly ash samples . Then fly ash was mixed with 2 %, 4 %, 8 %, 12 % , 16 % and 20 % of alkalis ( i. e. NaOH, KOH and $Ca(OH)_2$) as a percentage of total weight of the mixture and with the resultant mixture modified proctor test was performed , corresponding to particular maximum dry density ( MDD ) and optimum moisture content ( OMC ) obtained from heavy compaction test the unconfined compressive strength ( UCS ) samples and permeability samples were prepared . The UCS samples were cured for 0 , 3 , 7 , 28 and 70 days at a constant temperature of $27^0$C , whereas permeability samples are cured for 0 , 3 , 7 and 28 days before testing to evaluate the effectiveness of alkali stabilized fly ash as an alternate geo-material.The UCS samples were sealed with wax to avoid the loss of moisture and the permeability samples were made fully saturated before testing.

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## LIST OF ABBREVIATIONS

| Notation | Description |
|---|---|
| MDD | Maximum Dry Density |
| OMC | Optimum Moisture Content |
| UCS | Unconfined Compressive Strength |
| Cu | Co-efficient of Uniformity |
| Cc | Co-efficient of Curvature |
| G | Specific Gravity |
| K | Co-efficient Permeability |
| σ | Stress |
| ε | Strain |

# CHAPTER 1: INTRODUCTION

## 1.1 Introduction

A waste material produced from thermal power plants from burning of Pulverized coal is called fly ash. The burning of coal produces a fine residue which is carried in the flue gas and separated by electrostatic precipitators . This residue collected in a field of hoppers is called as fly ash . The fly ash can be disposed of in the dry form or in wet form in which it can also be mixed with water and disposed as slurry in ash ponds . Disposal of fly ash is one of the extreme challenges faced by the thermal power plants in India.

In several countries , including India , coal is the main fuel used in thermal power plants for power generation and fly ash is produced as a waste material from burning coal . Current annual production of coal ash is expected typically around 600 MT / year worldwide , out of which fly ash constitutes about 75 - 80 % of the total ash produced . Thus , the amount of fly ash generated has been increasing from thermal power plants throughout the world . Particularly in India the percentage utilization of fly ash is a mere of 5 %. Approximately 200 billion tonnes of coal ash reserves are in India and its annual production reaching 250 million tonnes roughly . The generation of fly ash increased about 131 million tonne during 2010 - 11 and is predicted to grow further.

In India coal based thermal power plants is a major source of power generation. Indian coal has high ash content which is in range of 30% to 50%. The quantity of Fly Ash produced depends onthe operating conditions of thermal power plants and the quality of coal used. The annual production of fly Ash presently in India is more than110 million tonnes and the land occupied by ash ponds is 65000 acre. Such a vast quantity does cause problems such as of land usage, health risks and environmental hazards. Both in disposal as well as in utilization, extreme care has to be taken for protecting the interest of human life and environment. When pulverized coal is burnt, the residue produced contains approximately 80% of Fly Ash and 20% of bottom ash.

Fig 1.1 Wet disposal of flyash

Fig 1.2 Dry disposal of flyash

### 1.2 Fly Ash: An Overview

Fly ash is a fine dusty material produced from burning coal during the generation of electricity in the thermal power plants. Fly ash consistsof mainly of silica, alumina and iron.Fly ash can be used for a portion of cement in the concrete because of the pozzolonic property, which improves the quality. The concrete which is generated with the use of flyash is denser in nature leading to smoother and tighter surface with less bleeding . Fly ash concrete provides an extraordinary architectural benefit with proficient textural consistency . Fly Ash can also be termed as Coal ash ,Pozzolana and Pulverized Flue ash.

### 1.3 Classification of Fly Ash

Depending upon the percentage of lime present and type of coal burmt fly ash can be classified into two types according to ASTM C618-03(2003a) as:

a) Class C.

b) Class F.

Class C fly ashes,also called as high calcium ashescommonly used in concrete industry because of it containing usually more than 15% CaO. Class C fly ashes have self cementitious property in addition of being pozzolanic in nature. It also has a presence of high calcium content which reacts readily with water even in the absence of lime.Class C fly ash is generally produced by burning lignite coal.

Class F type of fly ashcontains lower percentage of lime which is generated by burning bituminous coal or anthracite . Class F fly ashes are designated as low calcium ashes as they have calcium oxide (CaO) content less than 6%, , and are not self- cementing in nature but usually exhibit pozzolanic properties . Unburned carbon contentin these ashes is more than 2% and is found by loss on ignition (LOI) test . The major types of crystalline phases identified in fly ashes are Quartz , mullite and hematite , which are derivative of bituminous coal . Thus , major research regarding the usage of fly ash deal with Class F type as construction material.

The main objective of present study is to inspect the suitability of class F type of fly ash which contains low CaO content of about 1.4%asan alternative construction material or geo-material by stabilizing it using different alkalis. In civil constructions works, the fly ash can be utilized and this would enable to ease the disposal problems met by the thermal power stations and shortage of land as its properties are quite similar to the conventional earth material.

**1.4 Impact of Fly Ash on Environment**

Coal-based thermal power plants produced a huge volume of fly ash which may bring numerous problems for environment. These waste products are generally poisonous in nature , easily combustible , corrosive and reactive and cause damaging effects on the environment. The particles of fly ash ranging in size from 0.5 to 300 micron equivalent diameter , having light weight , has the potential to get airborne easily and contaminate the environment . If fly ash disposal is not managed properly in sea / rivers/ ponds can cause harm to aquatic life also . It can also pollute the under -ground water resources with traces of poisonous metals present in it. Generally , the thermal power plant dump the fly ash in the ash pond in the plant area,

called pond ash. If the fly ash is stabilized properly, it can substantiate to be valuable geo-material.Thus disposal of the fly ash appropriately is one of the chief concerns to be dealt with in the current generation. An innovative solution that would be efficient, effective and approved environmentally is required to overcome disposal problem. The wastes can be utilized as a construction material in highways, embankments etc. By utilizing fly ash as construction material we can get rid of another problem of crisis of good soil of preferred quality which is essential for civil engineering construction. The cost of good quality soil materials is also growing. Thus efficient and effective use of the fly ash as a substitute for the soil would not only aid to reduce the problem of disposal but also permit the organizations to preserve soil and decrease deforestation.

**1.5 Strength Characteristics of Fly Ash**

For promoting the use of fly ash as one of the leading construction material, it is desirable to improve and enhance some properties by stabilizing it by adding of some suitable additive like alkalis (i.e. NaOH, KOH and $Ca(OH)_2$). This project work aims at assessment of the usefulness of addition of alkalis in stabilizationof fly ash and its suitability to be used as a construction material. In this project, the fly ash used for experimentation was collected from the thermal power plant of CPP- NSPCL, Rourkela Steel Plant. Construction material implies two things first material over which any kind of construction can be done i.e. good quality conventional earth or replacement of it (Geo-material) and second material with which construction can be done i.e. concrete or replacement of it (Geo-material). For assessing the suitability of any construction material for various geotechnical engineering works its consistency limits , compaction characteristics , strength parameters and settlement value are the most important properties to be determined . In this project , the geo -engineering properties of fly ash and the stabilized fly ash with different percentage of alkalis was determined . The complete testing program was done in two phases. In the first phase , the physical and chemical characteristics of the fly ash samples were determined by conducting grain size distribution , hydrometer analysis , heavy compaction test , UCS test , falling head Permeability

test and pH test of original fly ash samples. In the second phase, fly ash was mixed with 2%, 4%, 8% ,12% , 16% and 20% of alkalis (i.e. NaOH, KOH and $Ca(OH)_2$) as a percentage of total weight of the mixture and with the resultant mixture modified proctor test was performed, corresponding to particular maximum dry density (MDD) and optimum moisture content (OMC) obtained from heavy compaction test the unconfined compressive strength (UCS) samples and permeability samples were prepared. The UCS samples were cured for 0, 3, 7, 28 and 70 days at a constant temperature of $27^0$C, whereas permeability samples are cured for 0, 3, 7 and 28 days before testing to evaluate the effectiveness of alkali stabilized fly ash as an alternate geo-material. The UCS samples were sealed with wax to avoid the loss of moisture and the permeability samples were made fully saturated before testing. X-ray Diffraction (XRD) was done for selected alkali stabilized fly ash samples to investigate the type of compounds formed due to the reaction between alkali and amorphous silica($SiO_2$) and alumina($Al_2O_3$) present in fly ash. Scanning Electron Microscope (SEM) and Energy Dispersive X-ray analyzer (EDX) studies were done for selected samples to study the surface morphology and presence of minerals in the alkali stabilized fly ash samples cured for particular days.

# CHAPTER 2: LITERATURE REVIEW

## 2.1 Introduction

In Indiacoal based thermal power plants constitute about 90% and it is predicted to be roughly around 200 billion metric tons. Hence, the installed capacity of production of electricity is 100,000 MW in our country and about 73% is generated by thermal power station. Overall there are 85 coal based thermal and other power plants in India. Low calorific value of 3,000–4,000 kcal/kg and high ash content of 35–50%are found in coals obtained in India . In order to accomplish the required energy generation, a large amount of coal is essentially required, producing more ash residue. Currently, India produces approximately around 100 million metric tons of ash; which is estimated to get doubled in the next decade. The wet method is the most widely used method adopted in India for ash disposal produced from coal burning. About one acre of land is required for 1 MW of capacity in this method n addition to a great capital investment. Therefore, the ash pondsapproximatelyconstitute about 26,300 hectares of plot in India. The use of fly ash in several industries was just meagre 3% in 1994, but after the realization ofconserving the environment in India the utilization of fly ash has gradually been increasing. In 1994, a Fly Ash Mission (FAM) was commissioned byIndian government with the main objective of developing confidence among the manufacturers for the disposal and application of fly ash, through projectsdemonstrated by technology. This Mission has till now selected 10 main areas and has commenced 55 demonstration of technological projects at 21 sites throughout India. The fly ash consumption has improved to almost 13% in 2002from 3% in 1994 which is still projected to grow. Anannouncement was issued by the Ministry of Environment and Forests department of the Government of India (MOEF 1999) in 1999, establishing the basic context for the improvement in consumption of fly ash and conserving environmental efforts. This notification directed the existing thermal power stations to attain 20% fly ash utilizationinside 3 years and 100% utilization inside 15 years. In India Fly Ash Mission had through technologically demonstrated projects implementedthis as one of themain areas of this mission. A few projects have been made and embankments have been built in India by utilization of pond ash (Vittal 2001). Guidelines for the application of fly ash in embankments (IRC 2001)had been published the Indian Road Congress. Fly ash for the reason of its self-hardening characteristics became a popular construction material. The nature of coal, degree of pulverization, furnace type used and temperature of firing control the variation in the properties of fly ash.

## 2.2 Literatures on Coal Ash and Its Geo-Engineering Properties

Many investigation have been carried out on the properties of coal ashes by various researchers to study their application as a construction material in various areas of civil Engineering. Some of them are precised below.

**Sherwood and Ryley (1970)**established that due to existence of free lime in fly ash, it exhibitsself-hardening characteristics.

**Gray and Lin (1972)** reported that many elements such as gradation, particle size and chemical composition etc affect the specific gravity of the coal ashes.

**Yudbir and Honjo (1991)** stated that due to presence of lime in flyash the self-hardening characteristic is enhanced. Unconfined strength as high as 20 MN/m2 in 28 days for some fly ash can be achieved depending on the free lime and carbon contents in the fly ash, while other fly ashes may attain strength 0.1-0.4 MN/m2 after16 weeks.

**Rajasekhar(1995)** stated that coal ash comprises of cenospheres and solid spheres . Low specific gravity of coal ash occurs due to the existence of huge number of hollow ceno-spheres grades from which the removal of entrapped air cannot be possible.

**Singh (1996)** stated the variation of unconfined compressive strength of fly ashes depends on the presence of free lime within them.

**Singh and Panda (1996)** found shear strength parameters at various water content and established that major part of the shear strength is because of internal friction.

**Pandian and Balasubramanian (1999)** explainedthat coefficient of permeability depends mainly on the particle size, amount of compaction and pozzolanic activity of fly ash. Higher value for coefficient of permeability is obtained for the bottom and pond ashes because of it being coarse grained and devoid of fines as compared to fly ash.

**Pandian(2004)** found that fly ash has angle of internal friction of about 30 degrees due to which it is a freely draining materialand has lower specific gravityleading to lower unit weights causing lower earth pressures. It can be stated that fly ash can be utilized in geotechnical applicationswith some modifications by adding additives to enhance its properties.

**Das and Yudhbir(2005)** studied the effect on geotechnical properties of fly ashes with lime content (Cao), iron content (Fe2O3), carbon content , morphology, and mineralogy of fly ash.

**Maitra et al. (2010)** studied the reaction between lime and compacted fly ash. Steam curing was used for curing compacted fly ash. They studied the kinetics of the reactions occuring and it was observed that curing conditions and additives significantlyaffectedthe reaction kinetics.

**Reddy and Gourav(2011)** studied that under steam curing at low temperature the characteristics of lime treated fly ash or by using an additives like gypsum give marginal increase in strength. Besides, the results showed that in normal curing conditions optimum lime–fly ash ratio was about 0.75 for yielding maximum strength and, 24 h of steam curingat 800 C is sufficient to attainapproximately possible maximum strength.

**Singh and Sharan (2013)** revealed thatstrength characteristics of compacted pond ash depends on compactive energy and degree of saturation. Here the (flyash / pond ash) sample was compacted to compactive energy ranging from 357 kJ/m3 to 3488 kJ/m3.out The optimum moisture content and maximum dry densities corresponding to different compactive energies was determined by conventional compaction tests. In this paper they delineates with a wrapping up that because properties of pond ash are similar to conventional earth materials. pond ash can replace the natural earth materials in geotechnical constructions.

### 2.3 Literature Review on Alkali Treated Fly Ash or Other Binders

Many investigative works have been done on the fly ash treated with alkalior other binder materials like meta-kaoline by the various researchers for studying their applicability in various field of civil Engineeringas a construction material. Some of theseare summarized below:

**Fernandez et al. (2004)** conducted a micro level study on a set of fly ash samplesactivated by alkali and thermally cured. The morphology of fly ash particles was investigated that can very well suit to real life situation. The fly ash was mixed with alkali activators and the resultant paste was cured for solidifying. Here the constituentof the fly ashwhich are glassy in nature gets transformed to compacted cement. The main aim was to finda conceptual model which is capable to describe the alkaline activationprocess of fly ash, free of the conditions of experiment.The results indicate that the amount of reaction keeps on increasing with time. But higher degree of reaction is attained during the first few hours of thermal curing.

**Fernandez- Jimenez et al. (2005)** conducted a study on alkaline activated fly ash mortars to study the relationship between their mineralogical and microstructural characteristics.The activation of fly ash allows for getting a material with comparable cementing features than that of OPC.In alkali activation of fly ashes, it is first mixed with some alkaline activators and then the composite mixture is cured. The constituentof the fly ash which are glassy in nature gets transformed to compacted cement.Theresults of the experiments shows that the product formed by the reaction is an alkaline aluminosilicategel, with crystalline structure. This product formed is responsible for the exceptional mechanical-cementitious properties of the fly ash activated with alkali.

**Puertas et al. (2000)** conducted a study on activation of fly ash / slag pastes with NaOHsolution and the nature of reaction products was investigated. The process parameters studied are alkali concentration, curing temperature and fly ash / slag ratios.fly ashes and Blast furnace slags are renownedconstruction materials and are used to manufacture blendedcements and concretes. Low hydration heat and high sulphate and water-sea resistanceare characteristics ofBlast furnace slag cements. Instead,fly ashes can be used aspozzolanic material to improve physical, chemical and mechanical properties of fly ash blended cements and concrete. The results shows that with increase in slag content in the pastes ,compressive strength increases and NaOH concentration developsthe strength.

**Frantisek- skvara et al.** conducted a study on aqueous suspensions of fly ash with activated alkali , fly ash mixed with ground granulated blast-furnace slag with activated alkali, fly ash mixed with blended Portland cement with activated alkali. The materials used in experimentation was power plant fly ash and ground granulated blast furnanceslag.The results obtained show that activation by alkali is capable of stimulating the reactivity of substances with the concealed chemical properties such as fly ash or combinations of fly ash with ground slag. Poor strength is exhibited even under hydrothermal curing by the chemically activated mixtures of fly ash. Again low 28 days strength is attained by the mixtures of a high fly ash content activated by a low concentration of alkali. Alsoa minimum content of slag in the ash slag mixture is required.for attaining high strength.

**F Purteas et al. (2002)** conducted a series of experiments to study the mineralogical and microstructural characteristics of alkali activated fly ash/ slag mixtures cured at different temperatures. The pastes obtained were tested by XRD, FTIR, MASNMR, SEM/EDX, atomic absorption and ion chromatography for its characterization, also the insoluble residue in HCl was determined.Here NaOH (chemical reagent) was used as an activator. Slag consisted largely of a glassy phase and crystalline phases were not detected by the X-ray diffraction analysis. Mechanical strengths werealmost same in both curing conditions by the results obtained.The results shows that at 28 days the mechanical strengths are higher in the pastes which were cured at 22 c than thoseobtained at 65 c. The existence of two diverse reaction products in the activated pastes have been proved by the results obtained.

**A Katz (1997)**conducted a study on mechanism ofactivation of fly ash and enhancement in the reactivity of fly ash blended cement was investigated.The investigation includes activating fly ash with strong base at different concentrations of alkali, temperatures and ratio of water to fly ash.The fly ash was obtained from local power station and a sodium hydroxide solution was immediately prepared before mixing with fly ash. The results indicate that the compressive strength increases with concentration of the solution for the same ratio of water to fly ash and curing temperature.Againdecreasing the sodium hydroxideto fly ash ratio by lowering theratio of water/fly ash, while maintaining theconcentration of solution constant it yielded a lower compressive strength.

**M Criado et al. (2007)** conducted a study on effect of soluble silica content on the mechanical and microstructural development of the cementitious materials produced due to the alkali activation of fly ash. In their study, fly ah was activated by4 diverse alkaline solutions with diverse soluble silica contents. Here type F fly ash was used. The fly ash was activated with different alkaline solutions, all with constant sodium oxide content (≈ 8%), but with different proportions of soluble silica. The results shows that at short curing times (8 hours), the development of high mechanical strength in the material is favoured withan increase in the soluble silica content.At slightly longer curing times (20 hours), a considerable increase was detected in the strength of alkali activated fly ash with a lesser silica content.Furthermorea consistently beneficial effect on the mechanical strengthwas obtained for longer curing times.

**A Fernandez- Jimenez et al. (2004)** conducted a study on the microscopic level of a set of alkali-activated fly ash and thermally cured fly ash samples and the microstructural development of fly ash-based cementitious geopolymers was investigated. In activation, the alkaline activators are used with fly ash and the resulting paste is cured to solidify. In this process,the constituentof the fly ash which are glassy in nature gets transformed to compacted cement.The result shows that the degree of reaction keeps on increasing with time. Moreover during the first few hours of the thermal curinghigh degree of reaction was achieved.

**Mo Zhang et al. (2013)** conducted a study on the feasibility of using geopolymer as the next-generation soil stabilizer. In thier study, a lean clay was mixed withmetakaolin based geopolymer at different concentrationto examine the capability of geopolymer in stabilizing soils. The soil to be studied was synthesized in the laboratory by mixing a soil collectedfrom a construction sitecampus and an ACTI-MIN CR kaolin clay at a dry mass ratio of 5:3.The results shows that compressive strength, failure strain and Young's modulus of the stabilized soil specimens increaseswith increase in concentrations of geopolymer, and shrinkage strains decreasesduring curing. The microstructural analyses established the formation of gels of geopolymer in the stabilized soil, and also the soil tends to form more homogeneous and compactmicrostructures after stabilization. Hence the studyexplains thatmetakaolin based geopolymer can be effectivefor stabilizing clayey soils.

### 2.4 Objective of the Project

Based on the review of the literature it was observed that the production of fly ashwill keep on increasing in coming years which needs large area to store which creates a problem for its safe economic disposal and causes environment hazards .A vast utilization of fly ash is only possible in civil engineering fields as a replacement to earth material as its properties are quite similar to that of the natural earth. To use the fly ash a replacement of earth or geo-material it is essential to stabilizing it using some proper stabilizing agent.

Keeping this in mind this project aims:

(i) To establish different types of alkali (i.e. NaOH, KOH and $Ca(OH)_2$) as a fly ash stabilizer.

(ii) To evaluate the effectiveness of alkali stabilized fly ash as alternate geo-material in civil engineering construction.

# CHAPTER 3: EXPERIMENTAL PROGRAMME

## 3.1 Introduction:

The chief concern for coal based thermal power stations is safe disposal of fly ash in an economical way. The difficulties faced by the thermal power stations for safe disposal of fly ash can be avoided by utilizing fly ash in construction industry as an alternate geo-material like in embankment, landfill, bases and sub-bases of a road and for any kind of super structure construction above it etc. Civil engineering field is gaining momentum in construction as it proves to be an efficient means of bulk application of waste material like flyash. But unstabilized fly ash didn't posses much strength to be used as a construction material. Thus to alter the waste material into reliable construction material, it is essential to improve some engineering properties of fly ash by stabilizing it using alkalis (i.e. $NaOH$, $KOH$ and $Ca(OH)_2$) to use it as a construction material . We can predict the behaviour of field structures from the laboratory test result pattern. This is used for understanding the structures in the field and can be used in developing mathematical relationship for practical purpose. In present study a series of modified proctor test, unconfined compressive strength test, falling head permeability test, pH test have been conducted to evaluate the effectiveness of alkali stabilized fly ash as alternate geo-material to use in civil engineering construction. Furthermore, XRD, SEM and EDX studies have been done for selected samples cured for particular period to investigate the chemical phenomenon responsible for strength gaining. The materials used, preparation of sample and testing procedure have been discussed in this chapter.

## 3.2 Experimental Arrangements

### 3.2.1 Materials Used

#### 3.2.1.1 Fly Ash

In this project Fly ash was used from the thermal power station of Rourkela steel plant (RSP), foreign and vegetative matters were separated by sieving through 1 mm sieve. The collected samples were oven dried at the temperature of 105-110 degree. After that the fly ash samples were kept in airtight container for subsequent use.

Fig.3.1: Fly Ash

#### 3.2.1.2 Alkalis

Three types of alkali have been used in this experiment i.e. sodium hydroxide (NaOH), potassium hydroxide (KOH) and calcium hydroxide ($Ca(OH)_2$), all the chemicals are of laboratory use quality. NaOH (97% pure) and KOH(84% pure) in pellets form and $Ca(OH)_2$(95% pure) in powder form were used. All the chemicals were brought from Rourkelamarket.

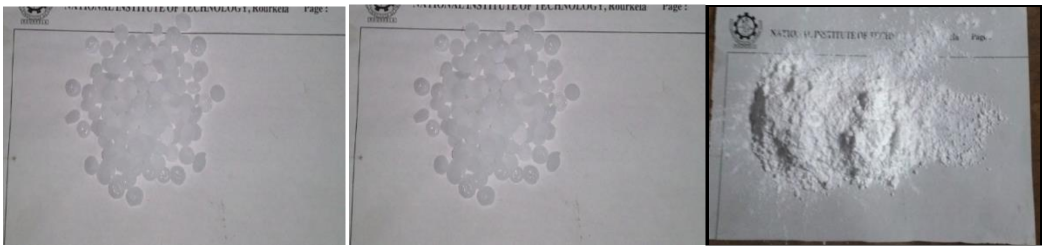


Fig.3.2: NaOH pellets, KOH pellets, $Ca(OH)_2$ powder

#### 3.2.1.3 Physical Properties of fly ash

The physical properties of the oven dried fly ash sample passing through 1mm sieve were determined and are presented in Tables 3.1.

Table 3.1 Physical Properties of fly ash

| Physical parameters | Values | Physical parameters | Values |
|---|---|---|---|
| Colour | Light grey | Shape | Rounded/sub-rounded |
| Silt &clay (%) | 88 | Uniformity coefficient, Cu | 5.67 |
| Fine sand (%) | 12 | Coefficient of curvature, Cc | 1.25 |
| Medium sand (%) | 0 | Specific Gravity, G | 2.38 |
| Coarse sand (%) | 0 | Plasticity Index | Non-plastic |

**3.2.1.4 Chemical Properties of fly ash**

The chemical characterization of the fly ash sample passing through 2mm sieve were determined by XRF study. Fly ash sample was made in the form of pressed powder for this. The results are given in Tables 3.2. and reults shows that the fly ash consists of mainly aluminum oxide and silicon oxide. Besides these two major constituents it contains iron oxide ($Fe_2O_3$), potassium (K2O), and titanium oxide ($TiO_2$).

Table 3.2 Chemical composition of fly ash

| Elements | F | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | $Fe_2O_3$ | ZnO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Composition (%) | 0.166 | 0.175 | 0.686 | 30.841 | 59.17 | 0.461 | 0.611 | 1.696 | 0.768 | 1.551 | 3.448 | 0.114 |

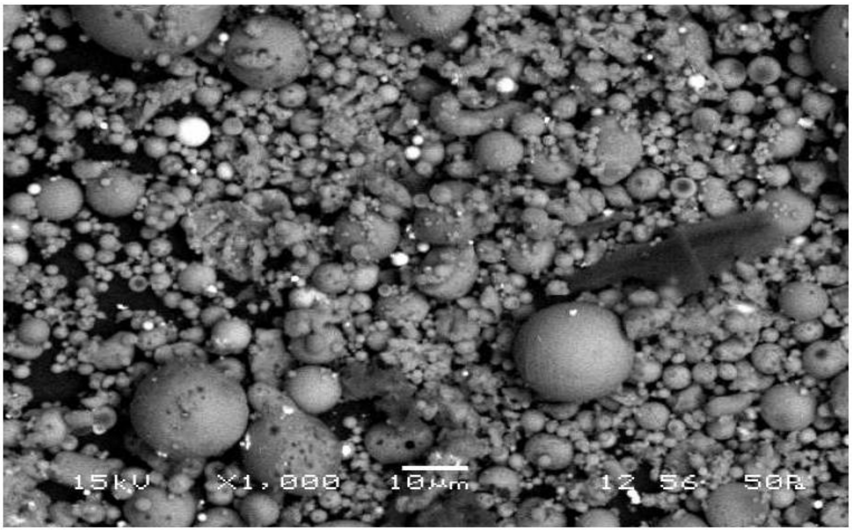



Fig.3.3: Scanning Electron Micrograph (SEM) of fly ash

Scanning Electron Microscope (SEM) was used to study the surface morphology of fly ash. The analysis shows that fly ash mostly contain rounded and sub-rounded particle and have uniform gradation. Micrographs were taken at accelerating voltages of 20 kV and X 1000 magnification for the best possible resolution.

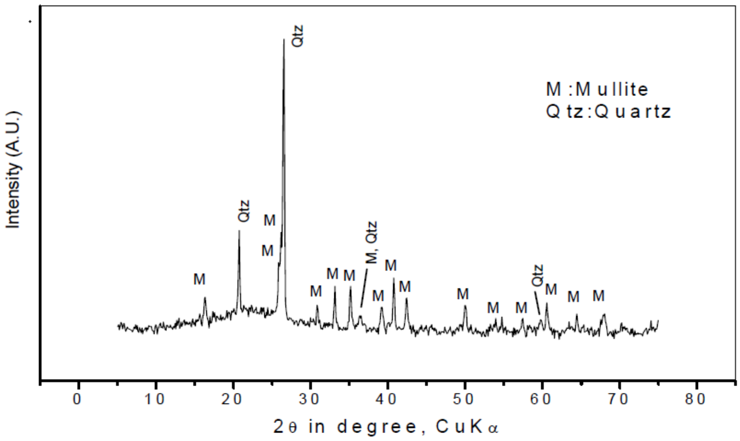



Fig. 3.4: XRD of fly ash

X-ray diffraction study was conducted of fly ash sample to find the main compound in it. From XRD study it was found that quartz and mullite is the dominating compounds in fly ash sample.

From XRF, XRD and SEM study of original fly ash sample it was concluded that the fly ash sample is a Class F type fly ash with lime (CaO) content less than 1.4%. Therefore it had less self hardening property and require stabilization.

### 3.3 Determination of Index Properties

#### 3.3.1 Determination of Specific Gravity

Le-Chatelier flask method with Kerosene as the solvent was used for determining the specific gravity of fly ash sample as per IS: 2720 (Part-III, section-1) 1980. The specific gravity of flyash was obtainedas 2.38.

#### 3.3.2 Determination of Grain Size Distribution

Sieve analysis was conducted for coarser particlesaccording to IS: 2720 part (IV), 1975 and hydrometer analysisfor finer particles was conducted according to IS: 2720 part (IV).88% of fly ash was passing through 75μ sieve. Therefore the grain size of fly ash ranges from size of fine sand to size of silt. Coefficient of uniformity (Cu) and coefficient of curvature (Cc) was obtainedas 5.67 & 1.25respectively, signifying uniformly graded samples. The grain size distribution curve of fly ash is as shown in Fig 4.1.

### 3.4 Determination of Engineering Properties

#### 3.4.1 Moisture Content Dry Density Relationship

Heavy compaction tests were done as per IS: 2720 (Part VII) 1980to find the relationshipbetween moisture content and dry density. Fly ash was mixed with varying percentage of alkalis (0%, 2%, 4%, 8%, 12%,16% and 20%) by the total weight of the mixture. Fly ash was thoroughly mixed with sufficient amount of water and the mixed sample was compacted in proctor mouldwith modified proctor rammer of 4.5 kg in five equal layers.the moisture content of the compacted mixture was determined as per IS: 2720 (Part 2) 1973. From the compaction curve obtained, optimum moisture content (OMC) and maximum dry density (MDD) were determined. Similarly heavy compaction tests were conducted with different percentage of alkalis ( 2%, 4%, 8%, 12%, 16%, 20%) and the corresponding OMC and MDD were determined. In case of NaOH and KOH particular percentage of alkali in terms of total weight of the mixture was mixed with the adequate amount of water to make viscous solution, and the solution was kept for at least 24 hours before mixed with fly ash for test programme. In case of $Ca(OH)_2$ as it was available in powder form so particular percentage of it directly added to fly ash and mixed thoroughly for 5-6 minutes then necessary amount of water was added thus the mixture was made for test programme. This was done to study the effect alkali and compactive energy on OMC and MDD on alkali treated fly ash samples. The compactive energy used in this test programme 2483 kJ/m3 of compacted volume. The test results are presented in Table 3.3.

Table 3.3.Compaction characteristics of flyash added with three types of alkalis

| Chemical Type | **content (%)** | **MDD (gm/cc)** | **OMC (%)** |
|---|---|---|---|
| NaOH | 0% | 1.29 | 30.46 |
| | 2% | 1.36 | 29.47 |
| | 4% | 1.39 | 26.77 |
| | 8% | 1.43 | 26.15 |
| | 12% | 1.49 | 23.95 |
| | 16% | 1.52 | 21.6 |
| | 20% | 1.554 | 19.81 |
| KOH | 0% | 1.29 | 30.46 |
| | 2% | 1.29 | 30.535 |
| | 4% | 1.34 | 27.47 |
| | 8% | 1.41 | 26.14 |
| | 12% | 1.46 | 21.95 |
| | 16% | 1.46 | 21.45 |
| | 20% | 1.525 | 21.285 |
| Ca(OH)2 | 0% | 1.29 | 30.46 |
| | 2% | 1.33 | 30.24 |
| | 4% | 1.336 | 30.99 |
| | 8% | 1.3 | 31.98 |
| | 12% | 1.3 | 30.09 |
| | 16% | 1.325 | 29.77 |
| | 20% | 1.33 | 29.90 |

### 3.4.2 Determination of Unconfined Compressive Strengths

To study the unconfined compressive strength characteristics of soil and soli like material the Unconfined compressive strength(UCS) test is most common test. The cylindrical test specimens of fly ash and alkali stabilized fly ash of size 50 mm in diameter and 100 mm in height (diameter : height = 1:2) were prepared for testing according to IS: 2720 (Part X)by compacting to their corresponding MDD at OMC with compactive energy 2483 kJ/m3. Specimens were coated with wax after prepared to avoid the moisture loss during curing period.This specimens were sheared at an axial strain rate of 1.25 mm/min till failure. The sealed specimens were cured at a constant temperature of $27^{0}$C for periods of 0, 3, 7, 28, and 70 days before testing. For each alkali content and curing period three alike specimens were tested and the average value was stated.

Fig.3.5: Prepared UCS samples of fly ash stabilized with different alkali content and cured at $27^{0}$C

Fig.3.6: Wax coated UCS sample

Fig.3.7: Before testing, During testing, After testing (UCS sample)

The UCS values of specimens were determined from stress vs strain curve and the failure stress and failure strain curve for 0%, 2%, 4%, 8%, 12%, 16%, 20% alkali content (NaOH , KOH, $Ca(OH)_2$) with 0, 3, 7, 28 and 70 days curing period is reported in the following tables 3.4, 3.5, 3.6.

Table 3.4: Unconfined compressive strength of for sodium hydroxide (NaOH) solution and fly ash mixture compacted at heavy compactive energy and cured at $27^0$C temperature

| NaOH Content (%) | 0 Days | | 3 Days | | 7 Days | | 28 Days | | 70 days | |
|---|---|---|---|---|---|---|---|---|---|---|
| | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε ( %) | σ (kPa) |
| 0 | 3 | 631.09 | 3.25 | 588.18 | 3.25 | 593.34 | 3 | 506.94 | 3.25 | 776.43 |
| 2 | 3 | 574.18 | 2.25 | 505.64 | 2.25 | 635.97 | 2.25 | 630.75 | 2.50 | 529.67 |
| 4 | 2.75 | 575.665 | 1.75 | 1152.6 | 1.75 | 1519.4 | 2 | 1672.3 | 1.75 | 2968 |
| 8 | 3 | 879.38 | 3.25 | 4798.3 | 3.5 | 5300.5 | 2.75 | 4382.3 | 3 | 9221 |
| 12 | 3.75 | 708.33 | 2.5 | 3457.6 | 3.75 | 6133.7 | 2.25 | 7630 | 5.25 | 24803 |
| 16 | 3.25 | 633.09 | 3 | 3305.3 | 2.5 | 5296.5 | 3.5 | 11303 | 3.75 | 14215 |
| 20 | 3 | 646.70 | 2.5 | 3719.6 | 3 | 7245.4 | 3.5 | 9763.9 | 2.75 | 7400 |

Table 3.5: Unconfined compressive strength of for potassium hydroxide (KOH) solution and fly ash mixture compacted at heavy compactive energy and cured at $27^0$C temperature

| KOH Content (%) | 0 Days | | 3 Days | | 7 Days | | 28 Days | | 70 days | |
|---|---|---|---|---|---|---|---|---|---|---|
| | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε( %) | σ (kPa) |
| 0 | 3 | 631.09 | 3.25 | 588.18 | 3.25 | 593.34 | 3 | 506.94 | 3.25 | 776.43 |
| 2 | 2.5 | 436.75 | 2.25 | 385.75 | 3 | 646.61 | 2.5 | 353.57 | 3 | 574.84 |
| 4 | 2.5 | 504.35 | 1.75 | 340.56 | 2.25 | 432.66 | 2.25 | 583.84 | 2.75 | 720.41 |
| 8 | 3.25 | 448.88 | 1.75 | 916.91 | 1.5 | 1181.88 | 2 | 1029.55 | 2.25 | 1725.8 |
| 12 | 2.25 | 364.89 | 2.25 | 2762.8 | 2.25 | 3596.8 | 1.75 | 4318.50 | 2.25 | 5144.3 |
| 16 | 2.5 | 710.22 | 3 | 2970 | 2 | 4113.7 | 3 | 7179.73 | 2.5 | 8607.1 |
| 20 | 1.25 | 755.90 | 2.25 | 3379.1 | 2.25 | 4392.9 | 3.25 | 10577.5 | 3.5 | 12614 |

Table 3.6: Unconfined compressive strength of for calcium hydroxide ($Ca(OH)_2$) solution and fly ash mixture compacted at heavy compactive energy and cured at $27^0$C temperature

| $Ca(OH)_2$ Content (%) | 0 Days | | 3 Days | | 7 Days | | 28 Days | | 70 days | |
|---|---|---|---|---|---|---|---|---|---|---|
| | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε (%) | σ (kPa) | ε ( %) | σ (kPa) |
| 0 | 3 | 631.09 | 3.25 | 588.18 | 3.25 | 593.34 | 3 | 506.94 | 3.25 | 776.43 |
| 2 | 3 | 441.84 | 3 | 1158.7 | 3.25 | 1589.1 | 3 | 1039.7 | 3 | 1401.14 |
| 4 | 3.25 | 532.09 | 3 | 1086.3 | 3.5 | 1502.6 | 2.75 | 1478 | 2.75 | 2569.45 |
| 8 | 3.5 | 622.12 | 3.25 | 1057.7 | 3.25 | 1480.7 | 2.75 | 3132.4 | 3.75 | 7141.88 |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 12 | 3.75 | 754.65 | 3.75 | 1555.2 | 3.25 | 1728.4 | 2 | 5444.7 | 4.25 | 15442.3 |
| 16 | 3.25 | 1660.3 | 2.25 | 2413.7 | 3.75 | 3141.3 | 4.25 | 11216 | 2.75 | 13471.9 |
| 20 | 2.75 | 1897 | 3 | 2802 | 2.75 | 3037 | 4.5 | 16504 | 2.5 | 14839.4 |

### 3.4.3 Determination of Co-efficient of Permeability

As fly ash is a fine grained material to determine its hydraulic conductivity falling head permeability test was conducted for original fly ash samples as well as alkali stabilized fly ash samples as per IS: 2720 (Part XVII)-1986. Test samples were prepared to their MDD at OMC in a permeability mould having diameter 10cm × height 12.5cm with a compaction energy of 2483 kJ/m3for evaluating hydraulic conductivity. However the prepared permeability stabilized fly ash samples were cured for 0, 3, 7, 28 days at humid environment to sustaining its moisture content for curing properly. Falling head permeability test was conducted and the coefficients of permeability for different alkali stabilized fly ash for different percentage and curing period were determined. Coefficient of permeability of these samples are presented in Table. 3.7, 3.8, 3.9 for NaOH, KOH and $Ca(OH)_2$ stabilized fly ash respectively.


Fig. 3.8: Permeability sample of stabilized fly ash with different alkali content kept for curing

Fig.3.9: Permeability sample during saturation before testing and during testing

Table 3.7: Co-efficient of permeability (k, cm/sec) of NaOH stabilized fly ash with different curing period at heavy compactive energy

| | K x 10^-6 (cm/sec) | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 0% NaOH | 83.7 | 67.2 | 39.1 | 13.1 |
| FA + 2% NaOH | 42.01 | 30.97 | 18.1 | 5.03 |
| FA + 4% NaOH | 23.2 | 18.32 | 9.03 | 1.98 |
| FA + 8% NaOH | 15.7 | 8.42 | 5.87 | 1.53 |
| FA + 12% NaOH | 7.87 | 3.83 | 1.32 | 0.88 |
| FA + 16% NaOH | 4.21 | 1.93 | 0.83 | 0.021 |
| FA +20% NaOH | 2.23 | 0.98 | 0.09 | 0.015 |

Table 3.8: Co-efficient of permeability (k, cm/sec) of KOH stabilized fly ash with different curing period at heavy compactive energy

| | K x 10^-6 (cm/sec) | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 0% KOH | 83.7 | 67.2 | 39.1 | 13.1 |
| FA + 2% KOH | 50.1 | 40.07 | 25.9 | 12.88 |
| FA + 4% KOH | 35.4 | 26.83 | 18.1 | 4.87 |
| FA + 8% KOH | 22.3 | 14.2 | 11.4 | 3.03 |
| FA + 12% KOH | 11.98 | 7.03 | 5.23 | 1.23 |
| FA + 16% KOH | 8.97 | 2.98 | 1.98 | 0.087 |
| FA +20% KOH | 5.98 | 2.23 | 0.63 | 0.038 |

Table 3.9: Co-efficient of permeability (k, cm/sec) of $Ca(OH)_2$ stabilized fly ash with different curing period at heavy compactive energy

| | K x 10^-6 (cm/sec) | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 0% $Ca(OH)_2$ | 83.7 | 67.2 | 39.1 | 13.1 |
| FA + 2% $Ca(OH)_2$ | 48.1 | 36.9 | 23.2 | 11.6 |
| FA + 4% $Ca(OH)_2$ | 29.2 | 22.7 | 14.4 | 3.67 |
| FA + 8% $Ca(OH)_2$ | 18.2 | 12.6 | 9.8 | 2.34 |
| FA + 12% $Ca(OH)_2$ | 9.81 | 5.87 | 4.74 | 1 |
| FA + 16% $Ca(OH)_2$ | 7.03 | 2.23 | 1.503 | 0.063 |
| FA +20% $Ca(OH)_2$ | 4.21 | 1.86 | 0.13 | 0.0253 |

### 3.4.4 Determination of pH

As per chemistry pH of an aqueous solution is taken out as negative log of hydrogen ion ($H^+$) activity. For soil and soil like material reaction is intimately associated with its pH.In this project pH test was conducted for alkali stabilized fly ash with varying alkali type, alkali content (2%, 4%, 8%, 12%, 16%, 20%) and curing period to determine the occurrence of pozzolanic type reaction between alkali and silica($SiO_2$) and alumina ($Al_2O_3$) present in the fly ash. The gel which was formed due to this reaction is responsible for strength gain in the alkali stabilized fly ash. The pH thet was conducted as per IS:2720 (Part- XXVI),1987. The values of pH of different samples are presented in table 3.10, 3.11, 3.12.

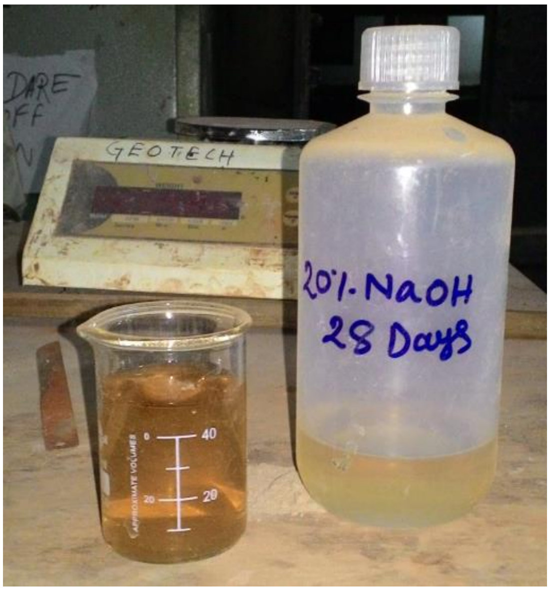



Fig.3.10: Prepared filtered sample for pH test

**Sample Preparation**

Small amount of sample (around 50 g)from the centre of UCS test specimens of alkali stabilized fly ash of different alkali content and curing period were oven dried at $105^0$C after they were tested for strength. After that oven dried samples were broken by using hammer into dust until the total dust samples was passed through 425 micron sieve. 30 gm sample from the dust was taken and mixed with 75 gm of distilled water (solid : liquid= 1:2.5). This mixture was stirred 3-5 times in one hour, then it was filtered by whatman 42 filter paper. The solution after filtration was tested for pH by using pH meter.

Table 3.10: pH of NaOH stabilized fly ash with different curing period

| | pH | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 2% NaOH | 10.29 | 10.08 | 9.92 | 9.7 |
| FA + 4% NaOH | 10.35 | 10.15 | 10.02 | 9.83 |
| FA + 8% NaOH | 10.58 | 10.42 | 10.28 | 10.05 |
| FA + 12% NaOH | 10.77 | 10.53 | 10.38 | 10.14 |
| FA + 16% NaOH | 11.38 | 11.22 | 11.05 | 10.64 |
| FA +20% NaOH | 11.97 | 11.83 | 11.68 | 11.23 |

Table 3.11: pH of KOH stabilized fly ash with different curing period

| | pH | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 2% KOH | 9.82 | 9.66 | 9.5 | 9.32 |
| FA + 4% KOH | 10.19 | 9.96 | 9.63 | 9.35 |
| FA + 8% KOH | 10.21 | 10.13 | 9.82 | 9.44 |
| FA + 12% KOH | 10.87 | 10.53 | 10.17 | 9.48 |
| FA + 16% KOH | 11.81 | 11.58 | 11.12 | 10.05 |
| FA +20% KOH | 12.32 | 12.17 | 11.85 | 10.89 |

Table 3.12: pH of $Ca(OH)_2$ stabilized fly ash with different curing period

| | pH | | | |
|---|---|---|---|---|
| Samples | 0 Days | 3 Days | 7 Days | 28 Days |
| FA + 2% $Ca(OH)_2$ | 10.68 | 10.51 | 10.23 | 9.78 |
| FA + 4% $Ca(OH)_2$ | 11.05 | 10.97 | 10.68 | 9.89 |
| FA + 8% | 12.07 | 11.93 | 11.77 | 10.22 |

| | | | | |
|---|---|---|---|---|
| $Ca(OH)_2$ | | | | |
| FA + 12% $Ca(OH)_2$ | 12.47 | 12.32 | 11.96 | 10.68 |
| FA + 16% $Ca(OH)_2$ | 12.78 | 12.57 | 12.16 | 11.02 |
| FA +20% $Ca(OH)_2$ | 12.97 | 12.78 | 12.53 | 11.95 |

**3.4.5. XRD, SEM and EDX Analysis**

**XRD:**

The X-ray diffraction (XRD) tests were used for characterization of the compounds formed due to the reaction between alkali and silica ($SiO_2$) and alumina ($Al_2O_3$) present in the fly ash at particular curing periods. This is performed by using Philips X' PERT System X-Ray diffractometer and shown in Figure.3.11 . 95% of solid materials are crystalline . When X-rays interact with a crystalline substance (Phase) , one gets a diffraction pattern . Every crystalline substance gives a pattern ; the same substance always gives the same pattern ; and in a mixture of substances each produces its pattern independently of the others (A. W. Hull, 1919). The powder sample was affixed to the sample holder and the upper surface of the sample was smeared by a glass slide to get a smooth and uniform surface . After that the specimen was then placed in the diffractometer for testing. It was observed that alkali alumino silicate type material were formed with a general formula of $M_n\{-(SiO_2)_z -AlO_{2}^{-}\}_n$ , where M was the alkali cation i.e. sodium ($Na^+$), potassium ($K^+$) and calcium ($Ca^{++}$). Generally, the defraction peak are reported in terms of the 2θ, where θ is the glancing angle of X-ray beam. The 2 θ values are then converted to lattice spacing 'd' in angstrom unit using Bragg's law. The range of 2θ value was given 3–75° with 0.05°/sec increments throughout .

Fig.3.11: XRD machine

**SEM:**

Scanning electron microscope investigate the particle surface morphology. These were done by a JEOL 6480LV SEM , equipped with an energy dispersive X-ray detector of Oxford data reference system as shown in Figure 3.12. A scanning electron microscope (SEM) produces images of a sample by scanning it with a focused electron beams. The electrons interacting with atoms in the sample produce several signals that can be detected and which contain information about the surface topography composition of sample. By using different condition and specimens, it is possible to obtain image showing the surface topography, surface potential distribution , magnetic domains , crystal orientations and crystal defects in specimen . SEM was done to study the change in the surface topography of alkali stabilized fly ash due to formation of alkali almino - silicate gel . The powdered as well as broken samples were loaded and fixed in the sample holder using a carbon tape which is further coated with a thin layer of electrically conductive platinum material . Micrographs were taken at accelerating voltage of 20 kV for the best possible resolution from the surface .


Fig.3.12: SEM machine

**EDX:**

Energy dispersive X - ray analyzer was used to characterize the mineral present in alkali stabilized fly ash samples . EDX makes use of the X - ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis.

**Sample Preparation**

Very small amount of sample (around 5-10 g) from the centre of UCS test specimens of alkali stabilized fly ash of different alkali content and curing period were oven dried at $105^0$C after they were tested for strength. After that oven dried samples were broken by using hammer into powder until the total powder sample was passed through 75 micron sieve. Then the sieved sample was mixed with acetone to stop the hydration process within the sample, as acetone got evaporate the sample was kept into air tight plastic envelop for XRD, SEM and EDX study.


Fig.3.13: Prepared sample for XRD, SEM and EDX

# CHAPTER 4: RESULTS AND DISCUSSION

## 4.1 General

A series of laboratory were carried out on fly ash specimens stabilized with different types and percentage of alkalis i.e. specific gravity test, grain size distribution, hydrometer test, heavy compaction test, unconfined compressive strength test, falling head permeability test, pH test and XRD, SEM and EDX study. The results obtained from these test aregiven and discussed in the present chapter.

## 4.2 Index Properties

### 4.2.1 Specific Gravity

Specific gravity of fly ash was determined as perIS: 2720 (Part-III, section-1),1980 and it was found to be 2.38 using Le-Chartelier method. The specific gravity of the fly ash was found lesser than earth materials and it depends on coal source, pulverization degree and temperature of firing. The presence of huge number of hollow cenospheres which contains the entrapped micro bubbles of air, or the deviation in the chemical composition, in particular iron contentleads to lower specific gravity.Generally the specific gravity of coal ash varies between 1.6 to 3.1. The specific gravity of resulting fly ash is mostly influencedby the presence of foreign materials in the fissures of the coal seams.

### 4.2.2 Grain Size Distribution

Fly ash consists of particles ranging from sand to silt as shown in figure 4.1. 88% of total fly ash sample was found to be passed through 75 micron sieve. The uniformity coefficient (Cu) and coefficient of curvature (Cc) for fly ash were found to be 5.67 & 1.25 respectively, which indicates that it was uniformly graded sample i.e. it consists of particles of about same size. The grain size distribution mostly depends on degree of pulverization of coal and firing temperature in boiler units and presence of foreign matters present in it.

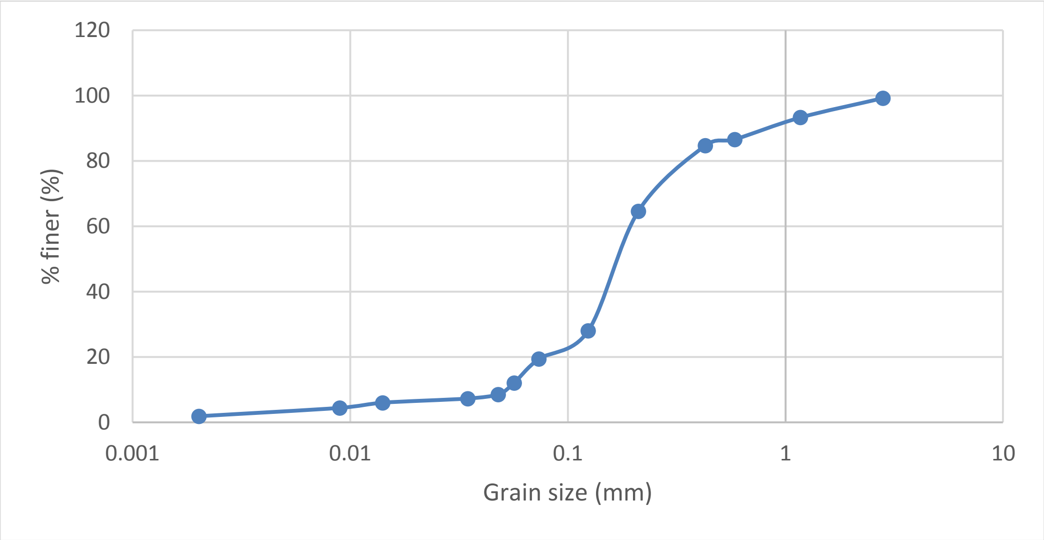



**Fig.4.1** Particle size distribution curve of fly ash

## 4.3 Engineering Properties

### 4.3.1 Compaction Characteristics

The heavy compaction characteristics of fly ash with different type and content of alkalis (i.e. NaOH, KOH, $Ca(OH)_2$) have been studied with compaction energy of 2483 kJ/$m^3$. The dry density (g/cc) and water content (%) relationship for different type of alkali have shown in the figures 4.2 to 4.4. The OMC (optimum moisture content ) and MDD (maximum dry density) variation with different type of alkali and alkali content have shown in figures 4.5 and 4.6. When fly ash was mixed with different percentage of sodium hydroxide (NaOH) and potassium hydroxide (KOH) solution, a steep increase in MDD and a steep decrease in OMC have been observed. As NaOH and KOH solutions are viscous and much lubricating than normal water, it coated the non-plastic fly ash particles; so during compaction as load was applied the coated fly ash particles sheared along each other surface resulting in within a small space more number of particles got adjusted and water which was in between two particles expelled out. Hence the maximum dry density increases and optimum water content decreases. On the other hand when fly ash was mixed with calcium hydroxide ($Ca(OH)_2$) powder and water was added separately, there was a slight increase in MDD and OMC but then almost there was not much alteration in MDD and OMC.As $Ca(OH)_2$ powder is also a non-plastic material like fly ash and the water is not as lubricant as NaOH or KOH solution resulting in almost no change on MDD and OMC have been observed.

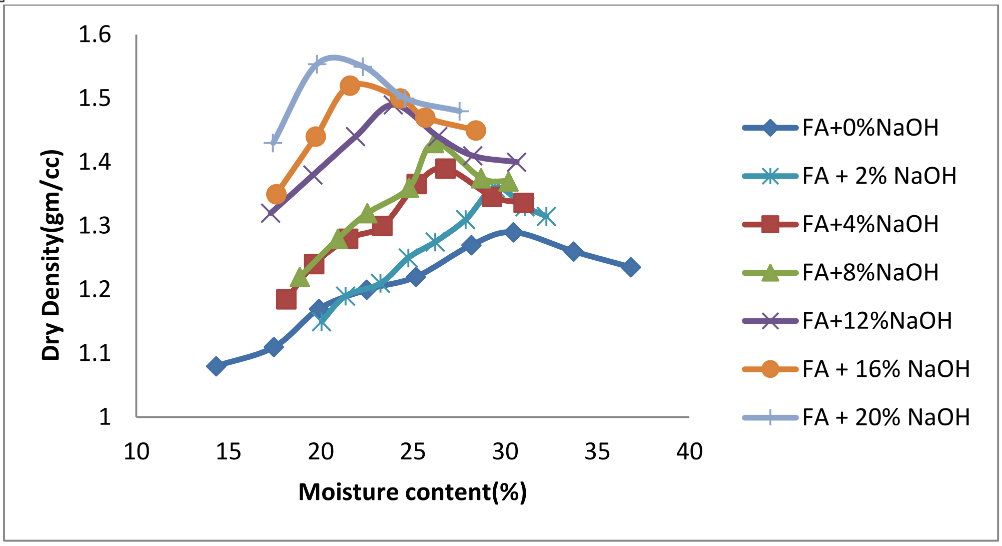



Fig.4.2: Variation of dry density (gm/cc) with moisture content(%) for fly ash mixed with NaOH

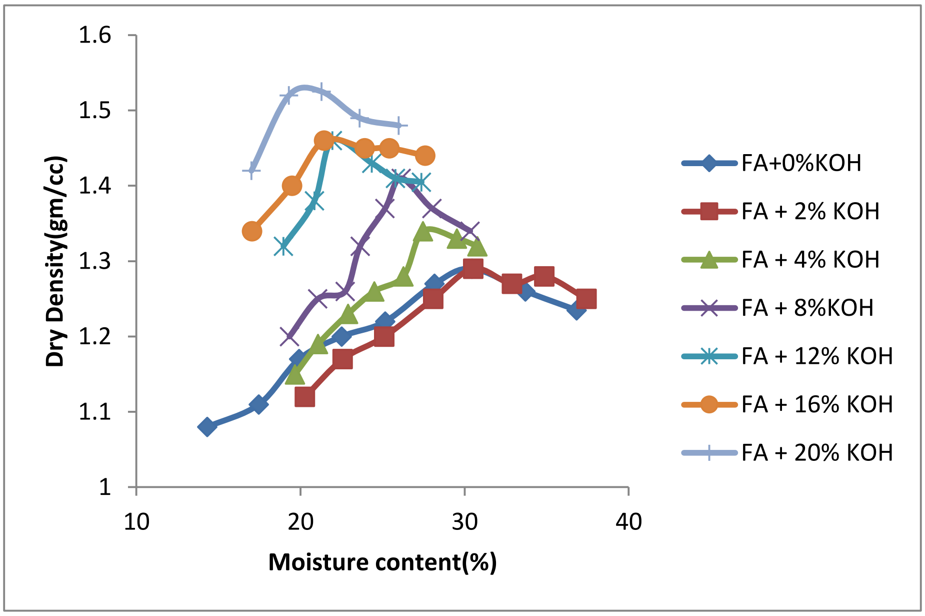



Fig.4.3: Variation of dry density (gm/cc) with moisture content(%) for fly ash mixed with KOH

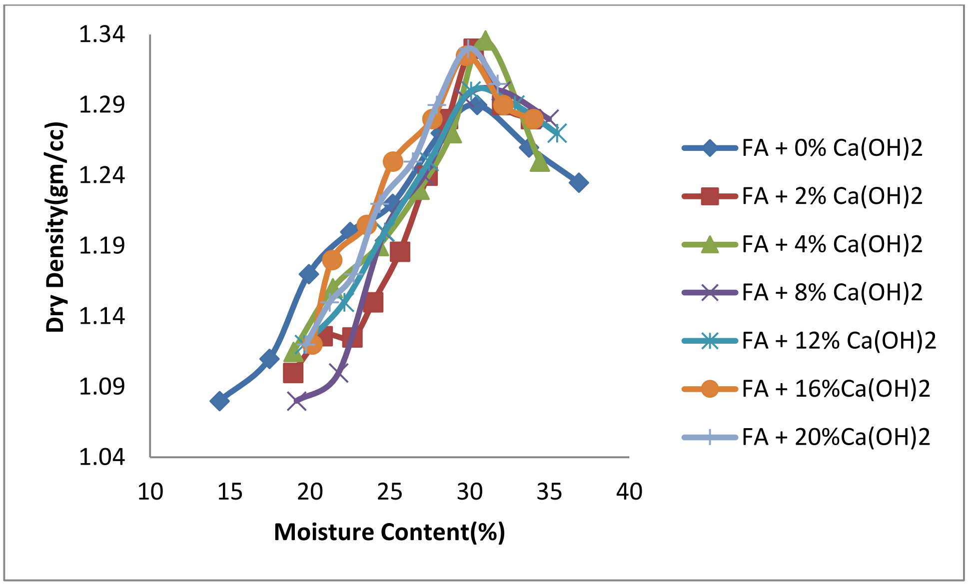



Fig.4.4: Variation of dry density (gm/cc) with moisture content(%) for fly ash mixed with $Ca(OH)_2$

For different NaOH content (0%, 2%, 4%, 8%, 12%, 16%, 20%) mixed with fly ash samples MDD increases from 1.29 gm/cc to 1.554 gm/cc and OMC decreases from30.46% to 19.81%.

For different KOH content (0%, 2%, 4%, 8%, 12%, 16%, 20%) mixed with fly ash samples MDD increases from 1.29 gm/cc to 1.525 gm/cc and OMC decreases from30.46% to 21.285%.

For different $Ca(OH)_2$ content (0%, 2%, 4%, 8%, 12%, 16%, 20%) mixed with fly ash samples MDD varies from 1.29 gm/cc to 1.336 gm/cc and OMC varies from 29.77% to 31.98%.

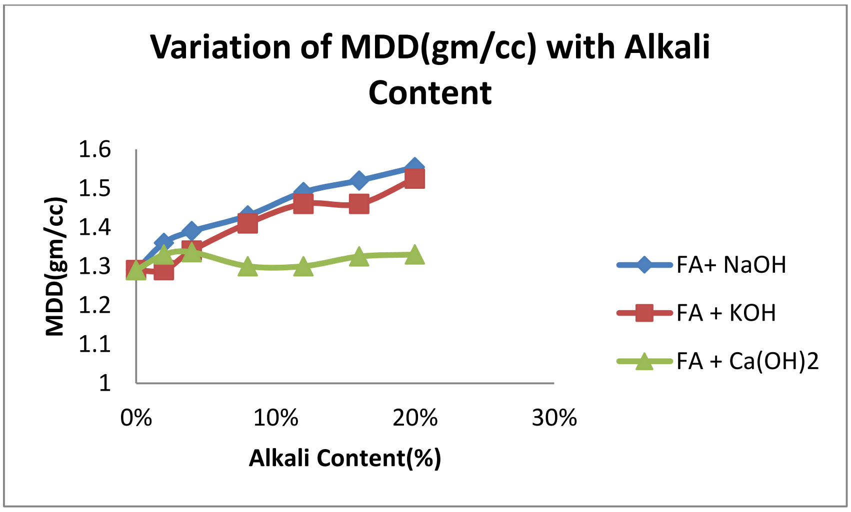



Fig.4.5: Variation of MDD (gm/cc) with alkali content for three types of alkalis

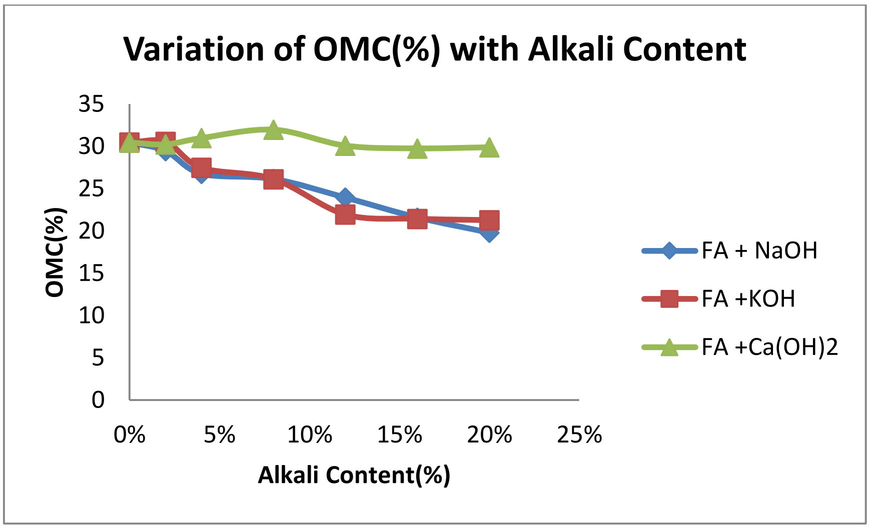



Fig.4.6: Variation of OMC (%) with alkali content for three types of alkalis

### 4.3.2 Determination of Unconfined Compressive Strength

Alkali treated fly ash specimens were prepared corresponding to their MDD and OMC for unconfined compressive strength test with heavy compactive effort as per IS: 2720 (Part- X), 1991. The stress – strain relationship of alkali stabilized fly ash with different alkali type, alkali content and curing period are presented in figures 4.7 to 4.15.

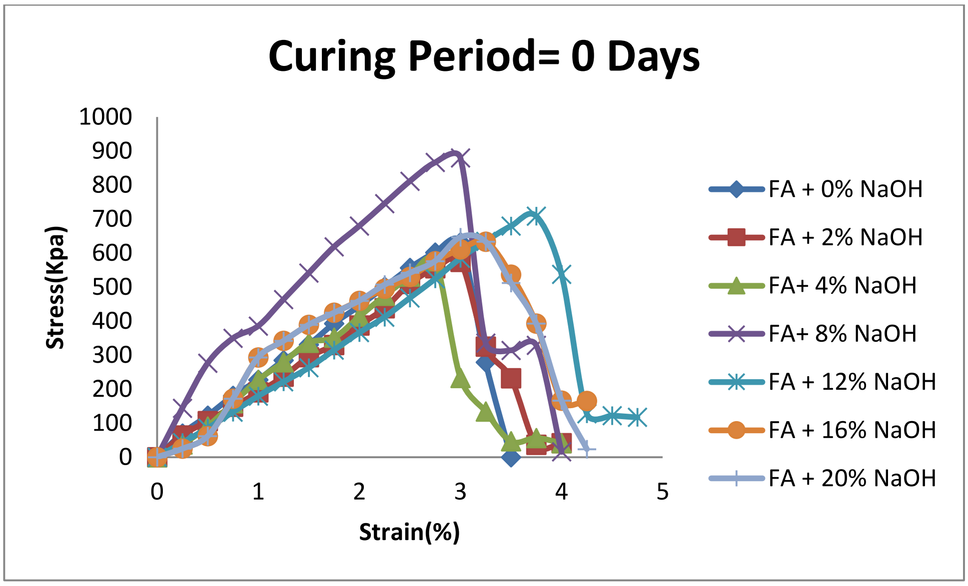



Fig.4.7(i)

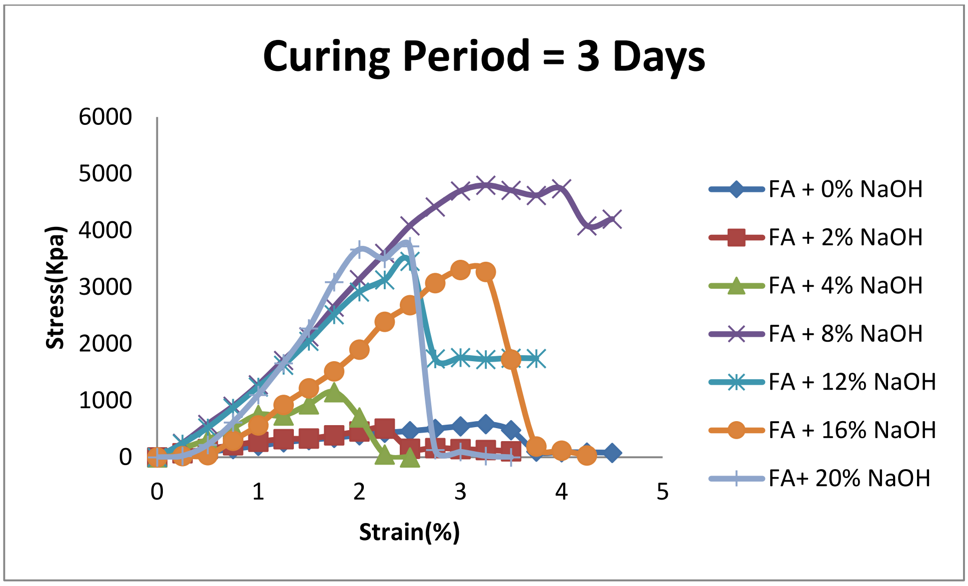
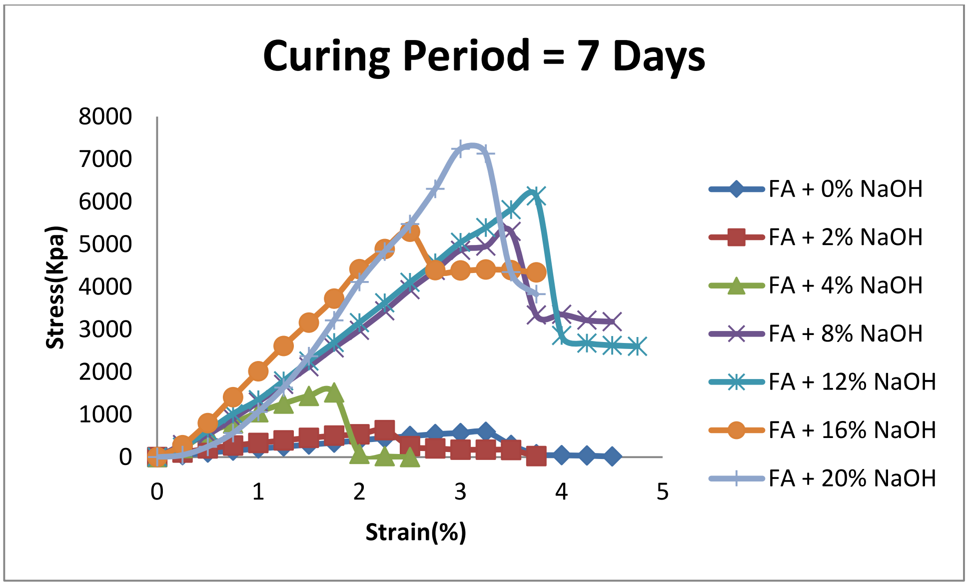



Fig.4.7(ii)

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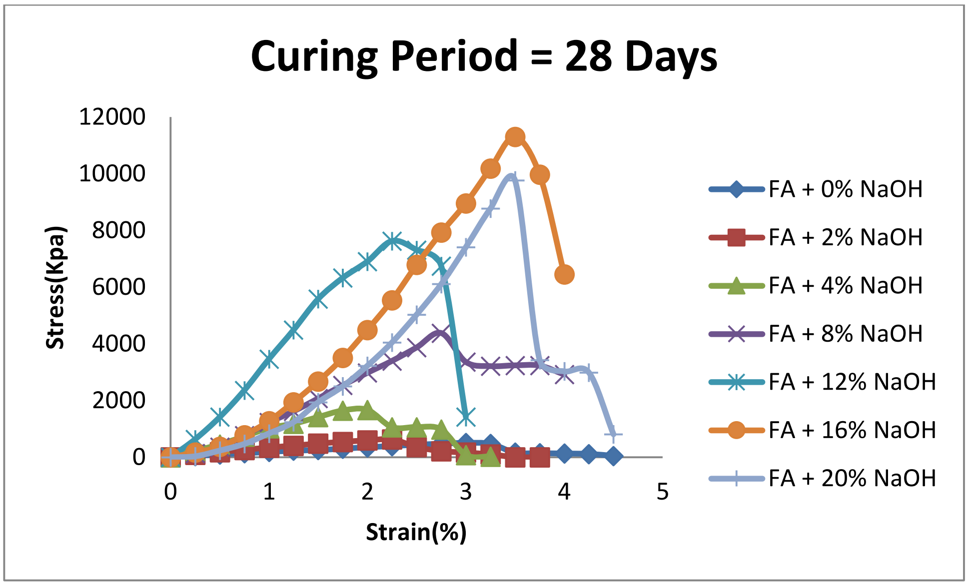



Fig.4.7(iv)

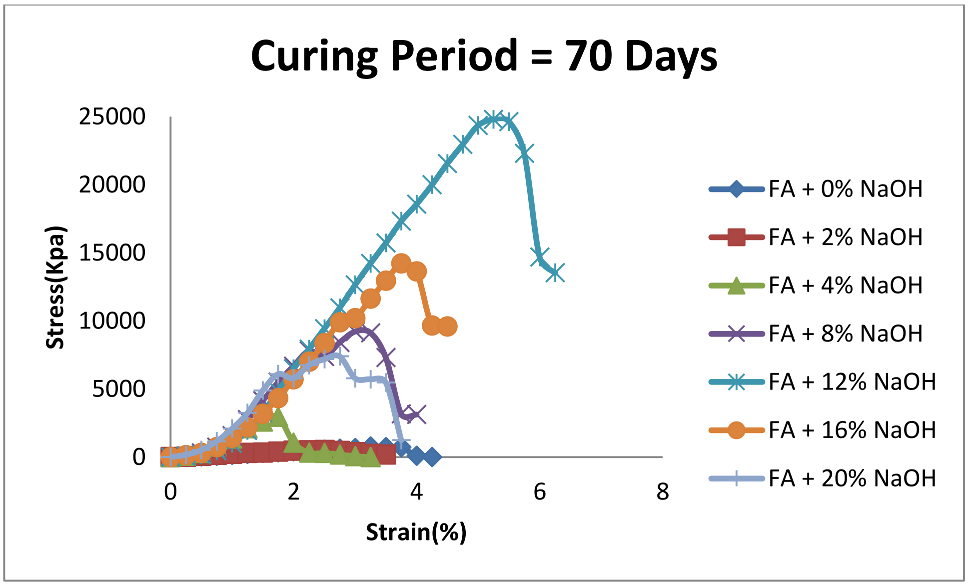



Fig.4.7(v)

Fig. 4.7(i) – 4.7(v): Stress – Strain relationship of NaOH stabilized fly ash at different curing period

From the above figures it is observed that the failure strain for NaOH stabilized fly ash varies between a wide range of 1.75% to 5.25% and the maximum failure stress obtained is 24803.5 Kpa for 12% NaOH , 70 days curing period. As the alkali content and curing period increases the failure pattern gets more brittle in nature.

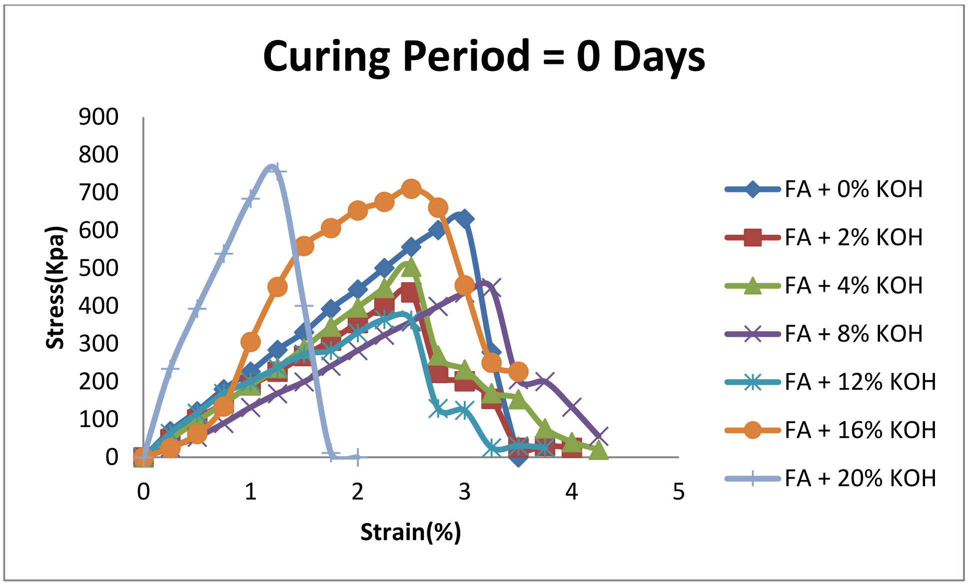



Fig.4.8(i)

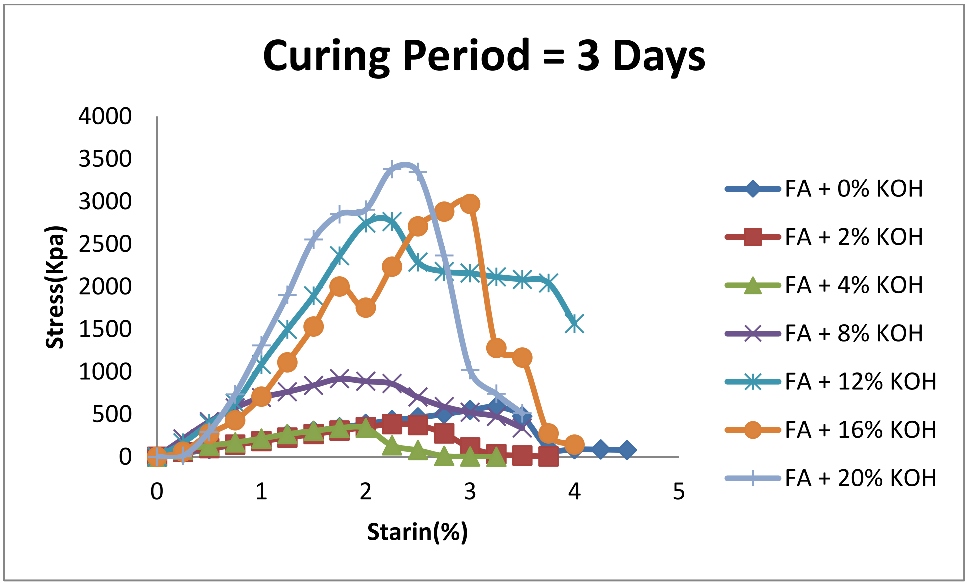



Fig.4.8(ii)

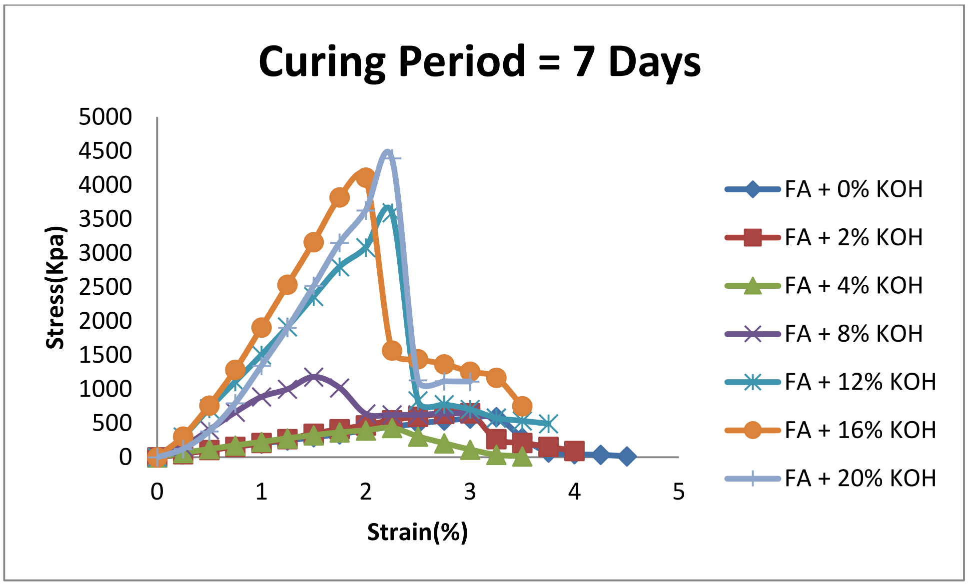



Fig.4.8(iii)

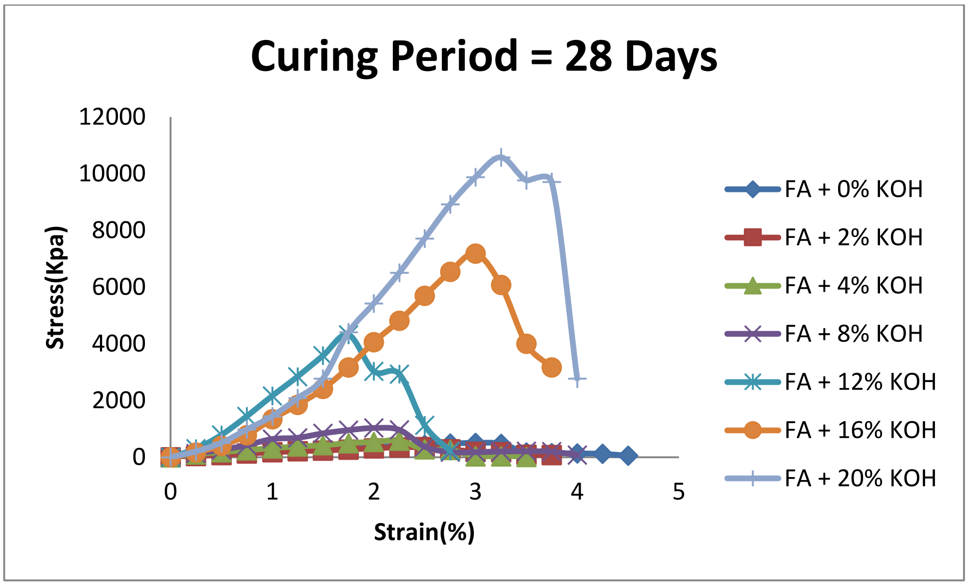



Fig.4.8(iv)

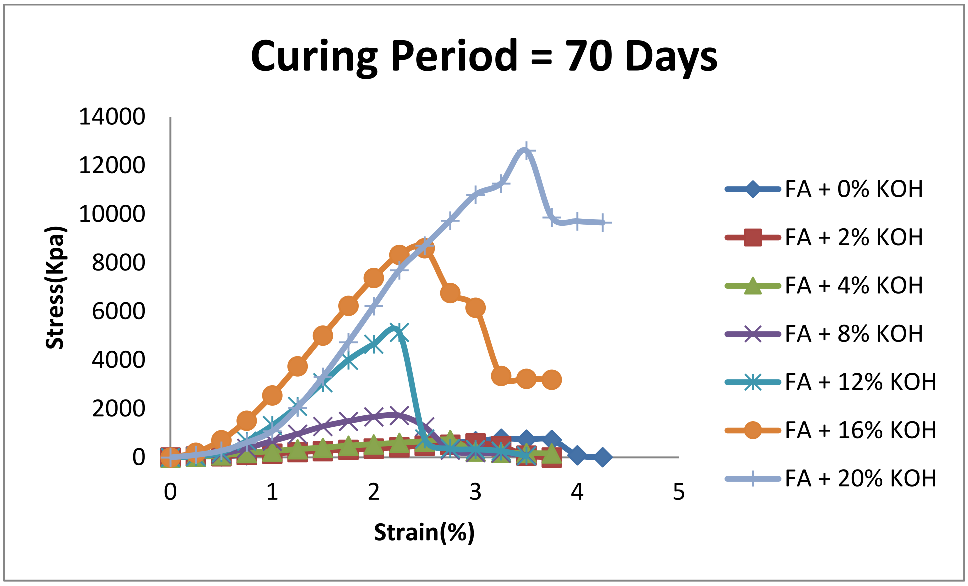



Fig.4.8(v)

Fig. 4.8(i) – 4.8(v): Stress – Strain relationship of KOH stabilized fly ash at different curing period

From the above figures it is observed that the failure strain for KOH stabilized fly ash varies between a wide range of 1.25% to 3.5% and the maximum failure stress obtained is 12614.44 Kpa for 20% KOH , 70 days curing period. As the alkali content and curing period increases the failure pattern gets more brittle in nature.

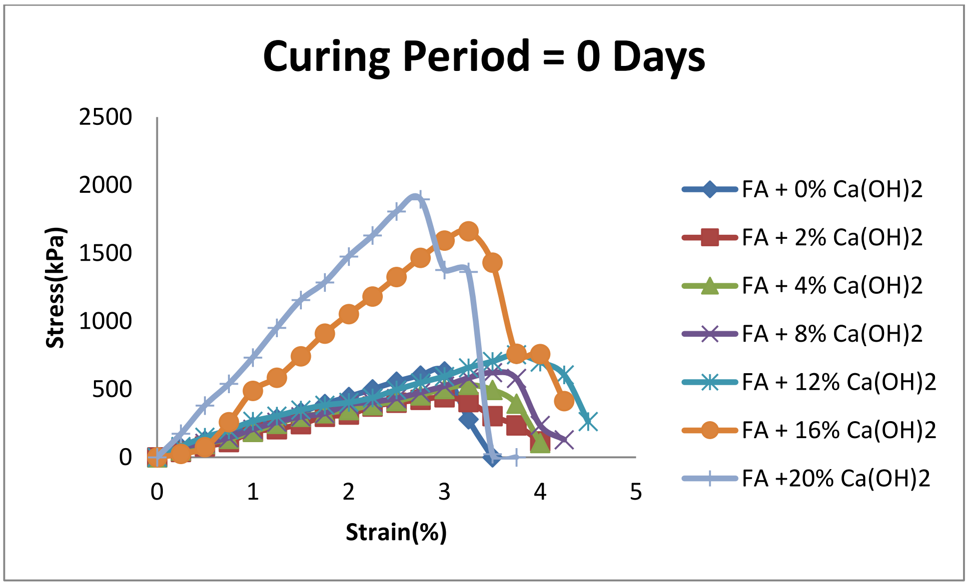
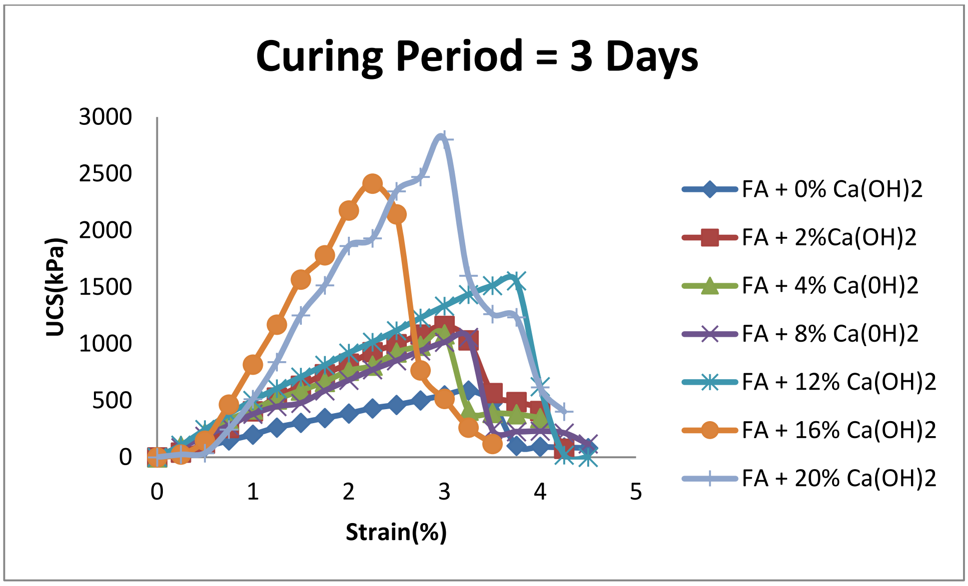



Fig.4.9(i)

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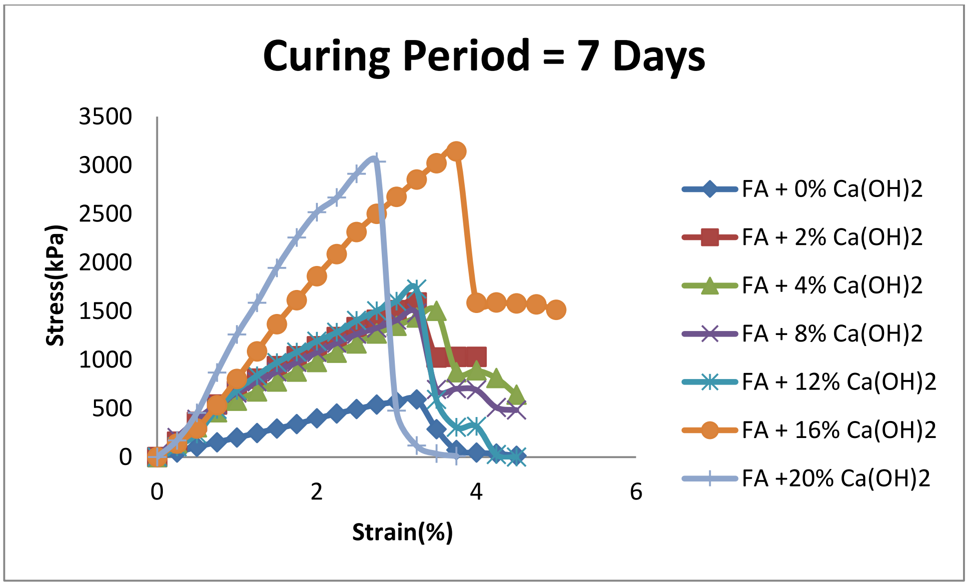



Fig.4.9(iii)

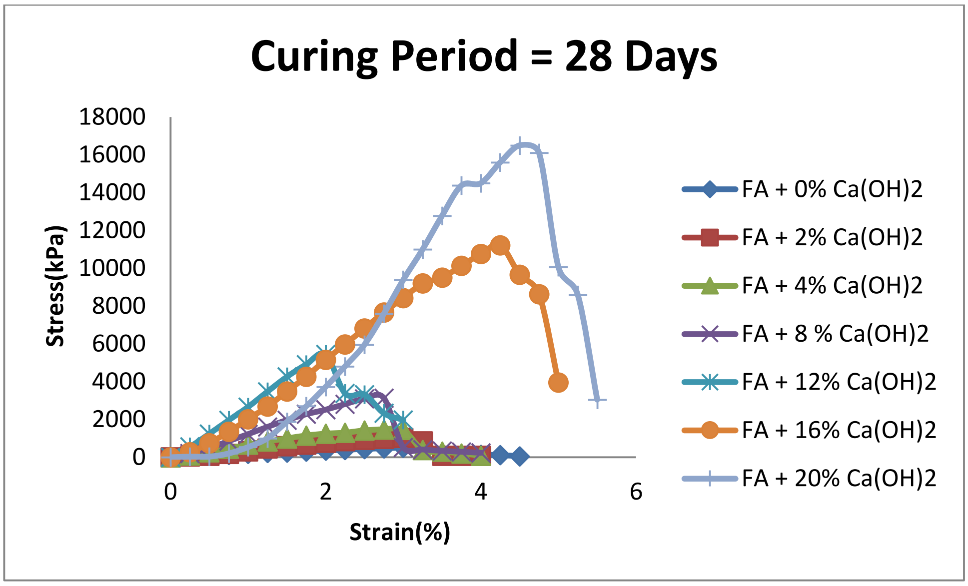



Fig.4.9(iv)

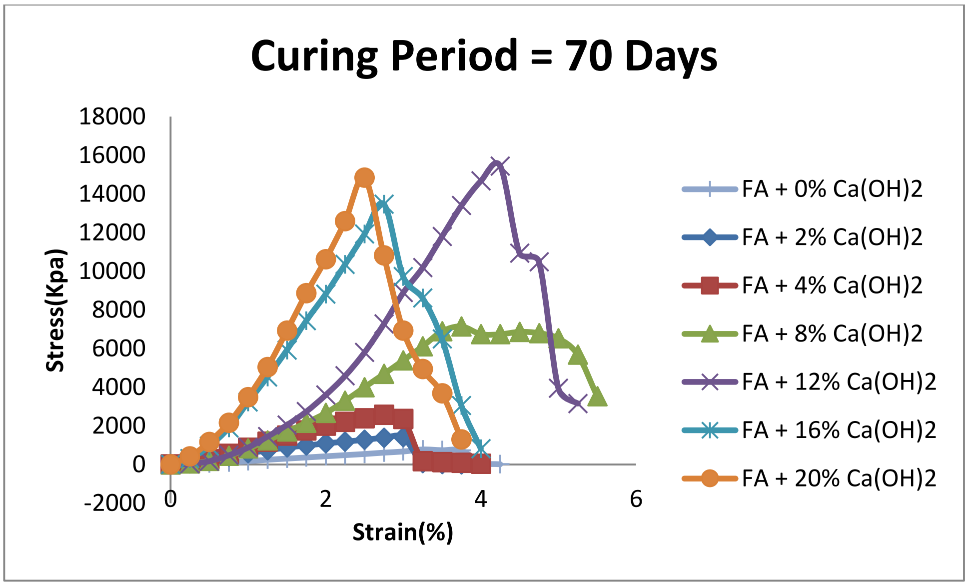



Fig.4.9(v)

Fig. 4.9(i) – 4.9(v): Stress – Strain relationship of $Ca(OH)_2$ stabilized fly ash at different curing period

From the above figures it is observed that the failure strain for $Ca(OH)_2$ stabilized fly ash varies between a wide range of 2% to 4.5% and the maximum failure stress obtained is 16504.45 Kpa for 20% $Ca(OH)_2$ , 28 days curing period. As the alkali content and curing period increases the failure pattern gets more brittle in nature.

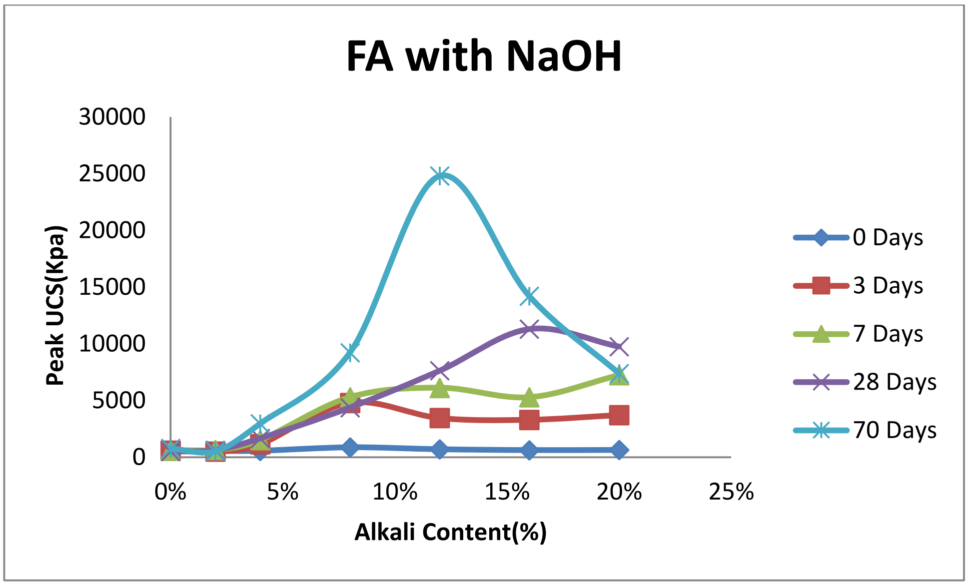



Fig.4.10: Variation of Peak UCS values with different NaOH content

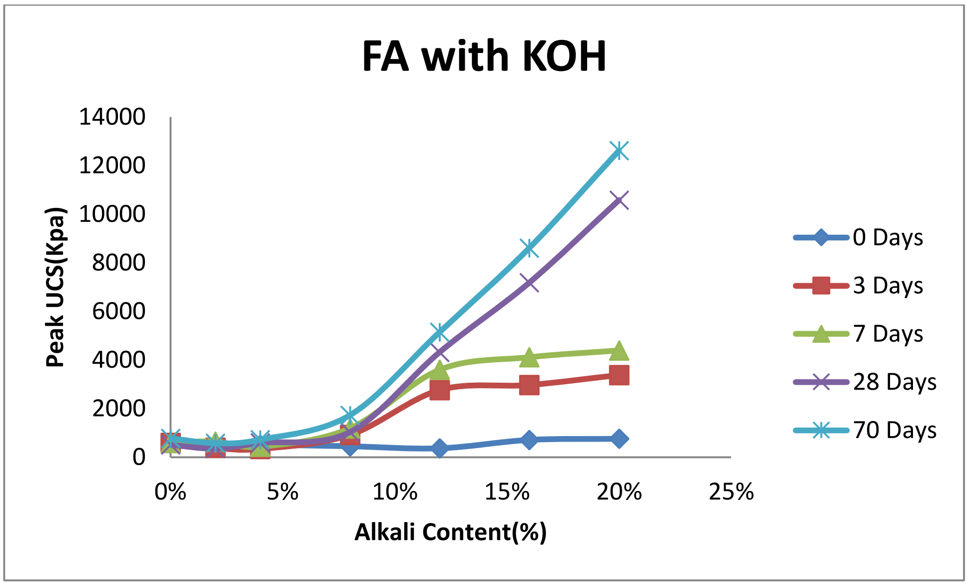



Fig.4.11: Variation of Peak UCS values with different KOH content

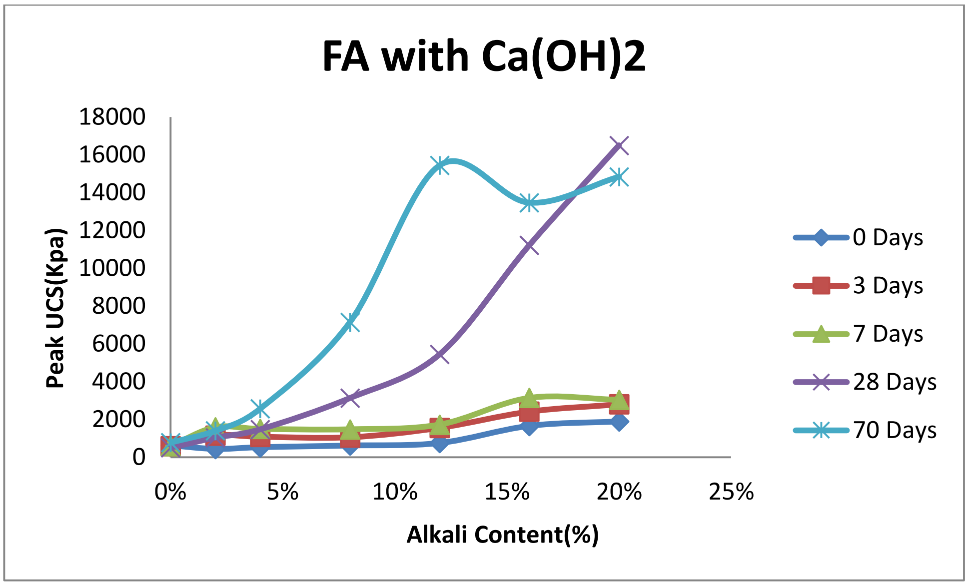



Fig.4.12: Variation of Peak UCS values with different $Ca(OH)_2$ content

On the other hand figure 4.10 to 4.12 represents the variation of peak UCS values with variation in alkali content. For NaOH stabilized fly ash at low alkali content(<12%) increment in UCS value is observed. As NaOH reacts with silica and alumina present in fly ash during the curing period and formed a gel like material called sodium alumino-silicate gel with a general formula $Na_n\{ -(SiO_2)_z -AlO_{2\,-} \}_n$. This gel filled the pore space in the UCS specimens and developed strength. But at higher alkali content(>12%) for 28 days and 70 days curing period a decrement in strength is observed this may because of excessive formation of gel. After filling up all the pore space in a particular UCS specimen if still gel remain there it can exert an outward pressure on the specimen which may results into development of crack so strength decreases. Though still investigation is going on this matter. Highest UCS value is obtained at 12% NaOH content.

For KOH stabilized fly ash at low alkali content(<8%) and curing period increment in UCS value is marginal. But as alkali content increases the UCS values increases steeply particularly at 28 days and 70 days curing period. KOH reacts with silica and alumina present in fly ash during the curing period and formed a gel like material called potassium alumino-silicate gel with a general formula $K_n\{ -(SiO_2)_z -AlO_{2\,-} \}_n$. This gel filled the pore space in the UCS specimens and developed strength. Highest UCS value is obtained at 20% KOH content.

For $Ca(OH)_2$ stabilized fly ash the variation of peak UCS is almost same like KOH stabilized fly ash, at low alkali content(<8%) and curing period increment in UCS value is marginal. But as alkali content increases the UCS values increases steeply particularly at 28 days and

70 days curing period. $Ca(OH)_2$ reacts with silica and alumina present in fly ash during the curing period and formed a gel like material called calcium alumino-silicate gel with a general formula $Ca_n\{ -(SiO_2)_z -AlO_{2\,-} \}_n$. This gel filled the pore space in the UCS specimens and developed strength. Highest UCS value is obtained at 20% $Ca(OH)_2$ content.

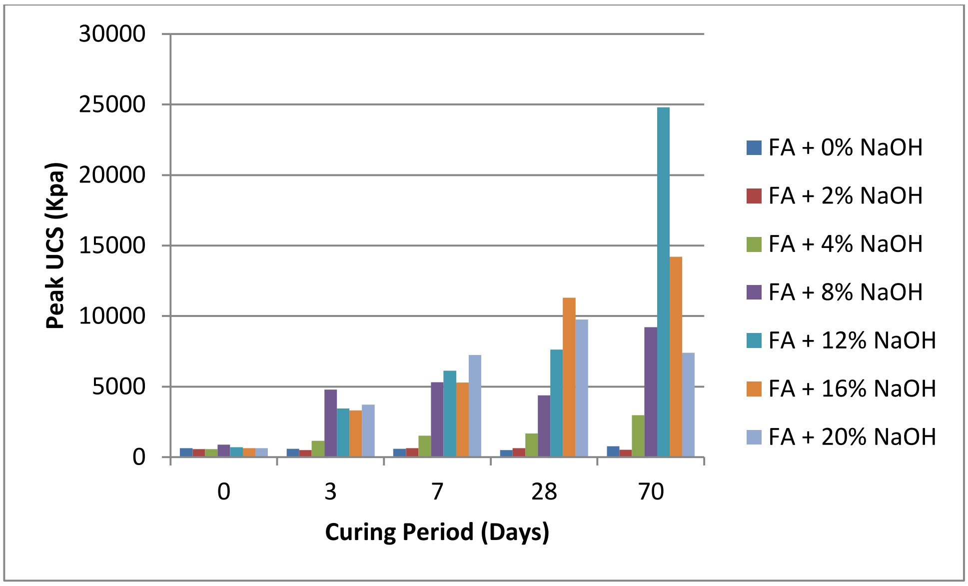



Fig.4.13: Variation of Peak UCS with curing period for NaOH stabilized fly ash

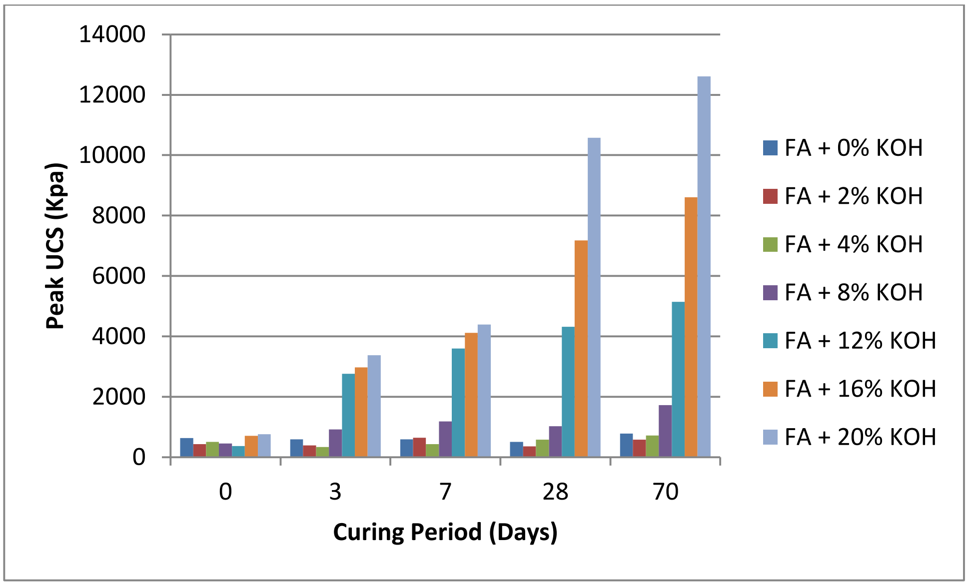



Fig.4.14: Variation of Peak UCS with curing period for KOH stabilized fly ash

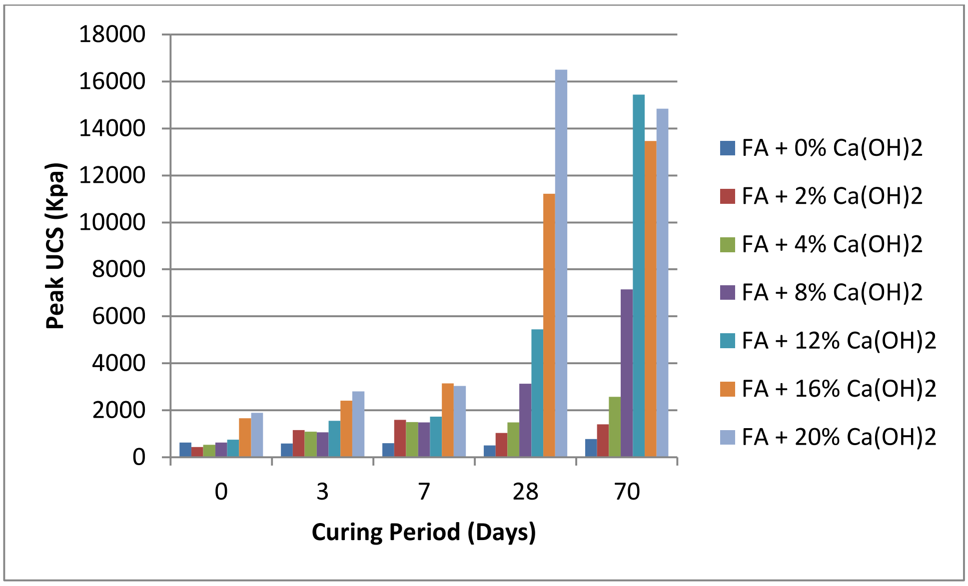



Fig.4.15: Variation of Peak UCS with curing period for $Ca(OH)_2$ stabilized fly ash

Figure 4.13 to 4.15 shows the variation of peak UCS values with curing period. For NaOH and KOH stabilized fly ash we observed highest UCS value at 70 days curing but for $Ca(OH)_2$ stabilized fly ash surprisingly some different phenomenon has been observed. For $Ca(OH)_2$ stabilized fly ash sample highest UCS value is obtained after 28 days curing. This may be because of the excess amount of calcium almino-silicate gel which formed after 28 days of curing trying to exert crack on the UCS specimen resulting in reduction in UCS value at 70 days curing period.

### 4.3.3 Determination of Co-efficient of Permeability:

Alkali treated fly ash specimens were prepared corresponding to their MDD and OMC for falling head permeability test with heavy compactive effort as per IS: 2720 (Part- XVII), 1986. The variation of co-efficient of permeability(k) with curing period and alkali content for three different types of alkalis is shown in figures 4.16 to 4.18.

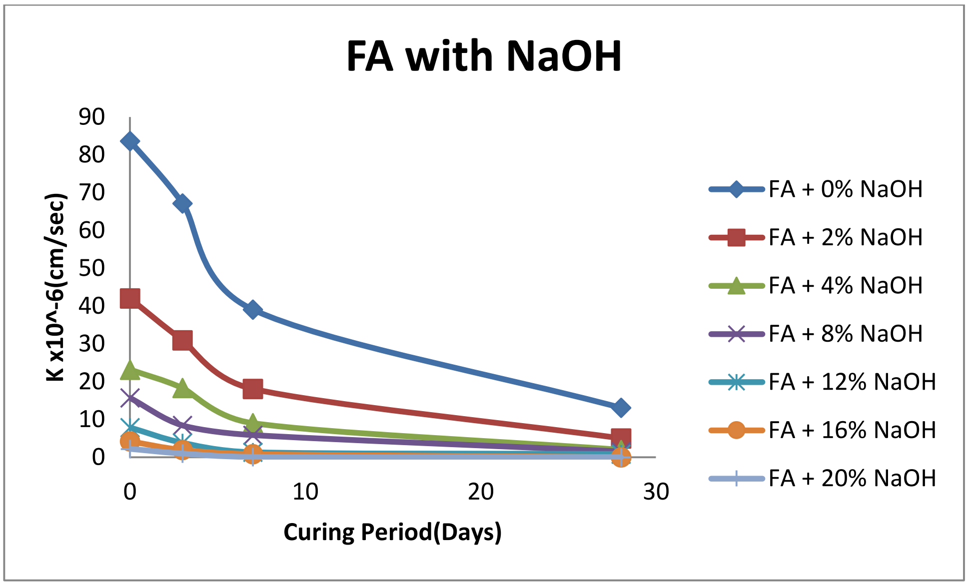



Fig.4.16 (i): Variation of k with curing period for NaOH stabilized fly ash

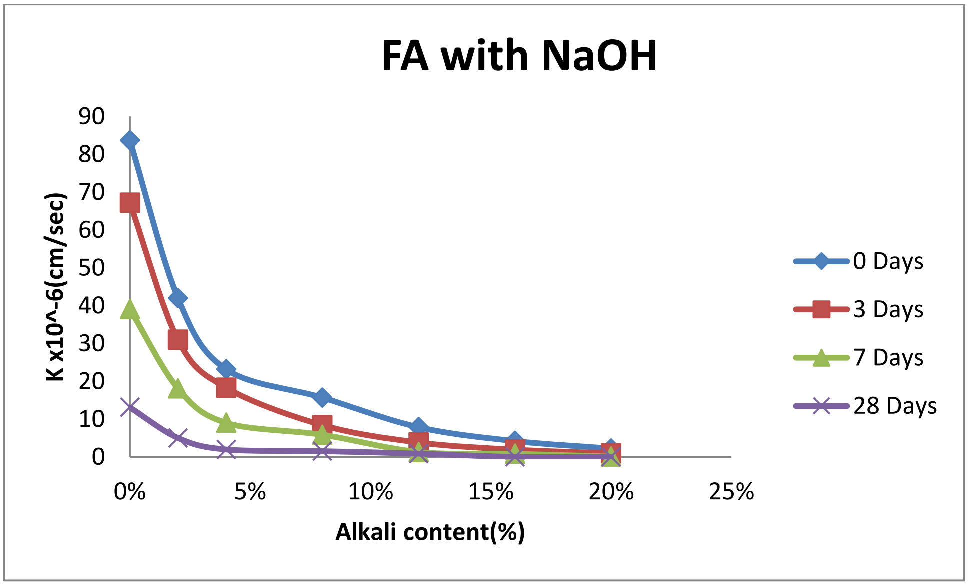



Fig.4.16 (ii): Variation of k with alkali content for NaOH stabilized fly ash

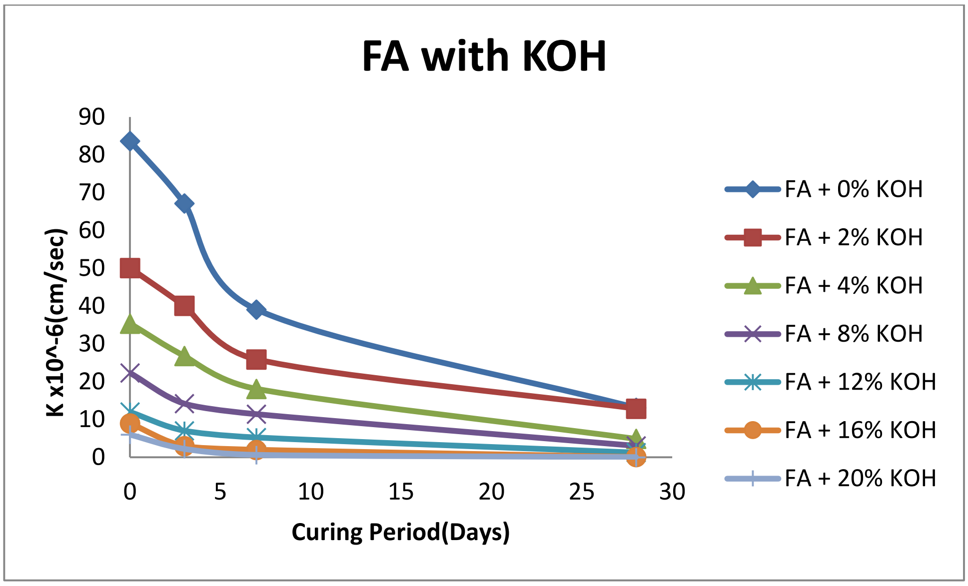



Fig.4.17 (i): Variation of k with curing period for KOH stabilized fly ash

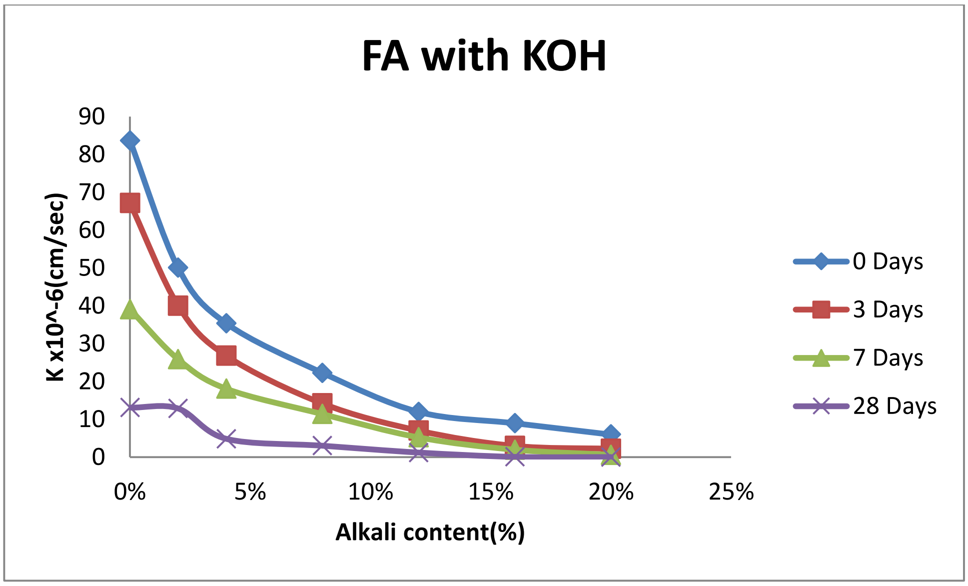



Fig.4.17 (ii): Variation of k with alkali content for KOH stabilized fly ash

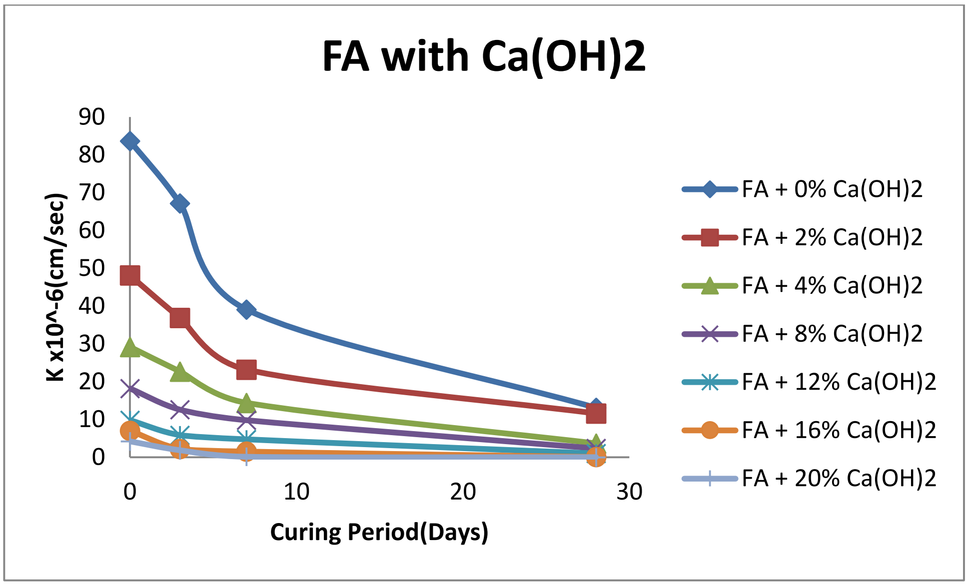



Fig.4.18 (i): Variation of k with curing period for $Ca(OH)_2$ stabilized fly ash

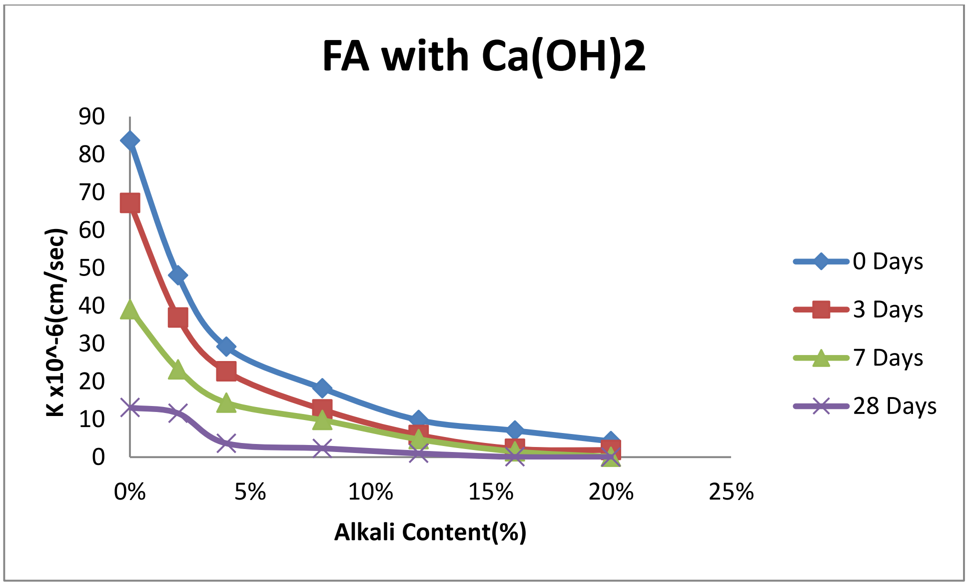



Fig.4.18 (ii): Variation of k with alkali content for $Ca(OH)_2$ stabilized fly ash

From above figures it is observed that the co-efficient of permeability (k) is continuously decreasing with curing period and alkali content for all three types of alkalis because the alkali alumino-silicate gel which is formed due to the reaction between alkali and silica and alumina filled up the pores in the permeability sample and solidify the total material. For NaOH, KOH and $Ca(OH)_2$ stabilized fly ash at 28 days curing the values of k obtained are 1.5 x 10^-8, 3.8 x 10^-8 , 2.53 x 10^-8 respectively.

### 4.3.4 Determination of pH

The pH test is conducted as per IS:2720 (Part- XXVI),1987 for alkali stabilized fly ash with varying alkali type (i.e. NaOH, KOH and $Ca(OH)_2$), alkali content (2%, 4%, 8%, 12%, 16%, 20%) and curing period to determine the occurrence of pozzolanic type reaction between alkali and silica($SiO_2$) and alumina ($Al_2O_3$) present in the fly ash. The alkali alumino-silicate type gel which is formed due to this reaction is responsible for strength gain in the alkali stabilized fly ash. The changes in pH value for alkali treated fly ash samples with curing period and alkali content are presented in the tables 4.19 to 4.20.

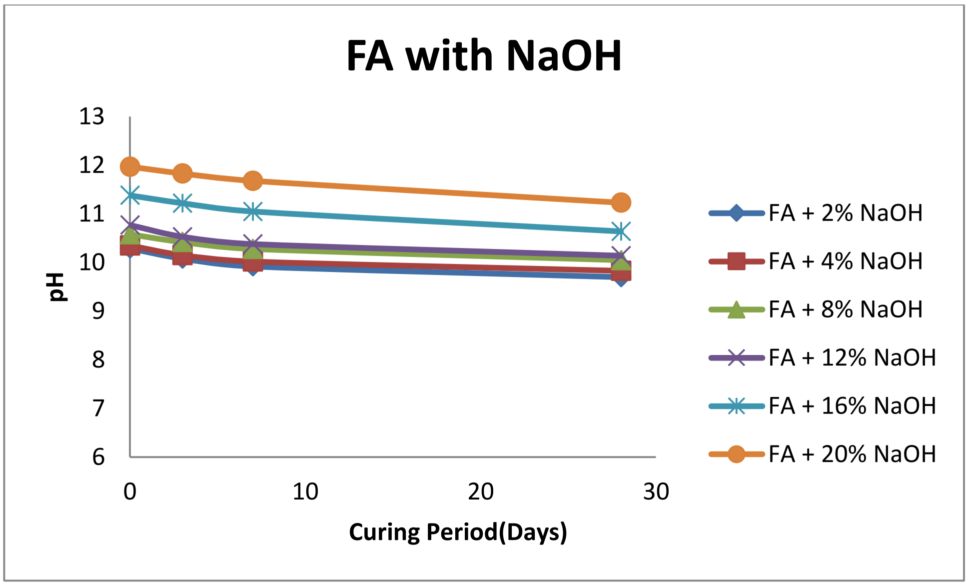



Fig.4.19 (i): Variation of pH with curing period for NaOH treated fly ash

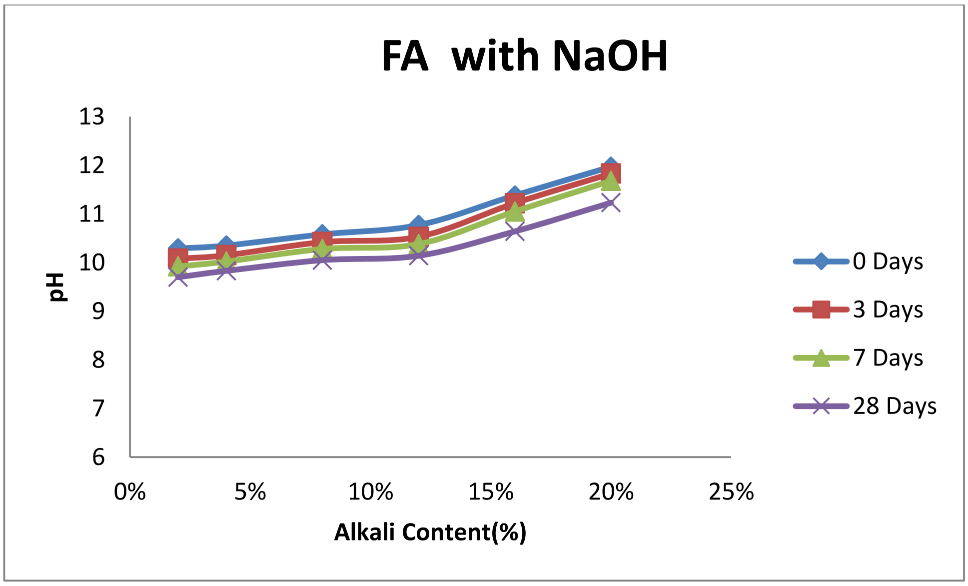



Fig.4.19 (ii): Variation of pH with alkali content for NaOH treated fly ash

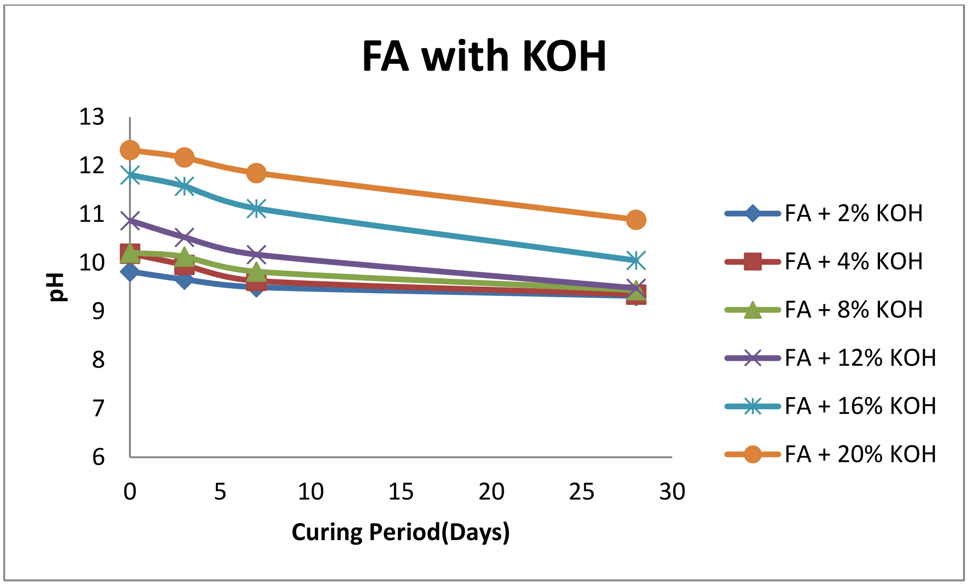



Fig.4.20 (i): Variation of pH with curing period for KOH treated fly ash

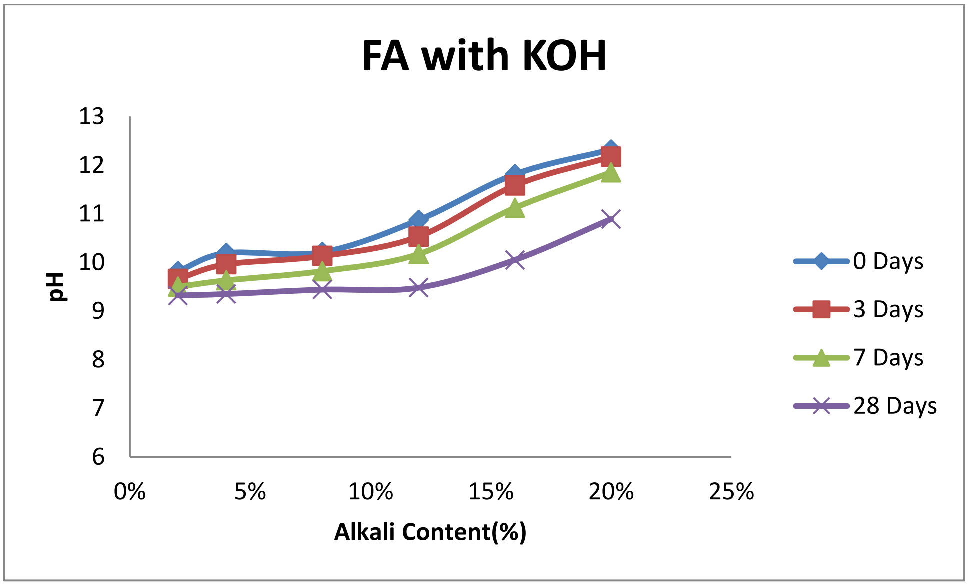



Fig.4.20 (ii): Variatioon of pH with alkali content for KOH treated fly ash

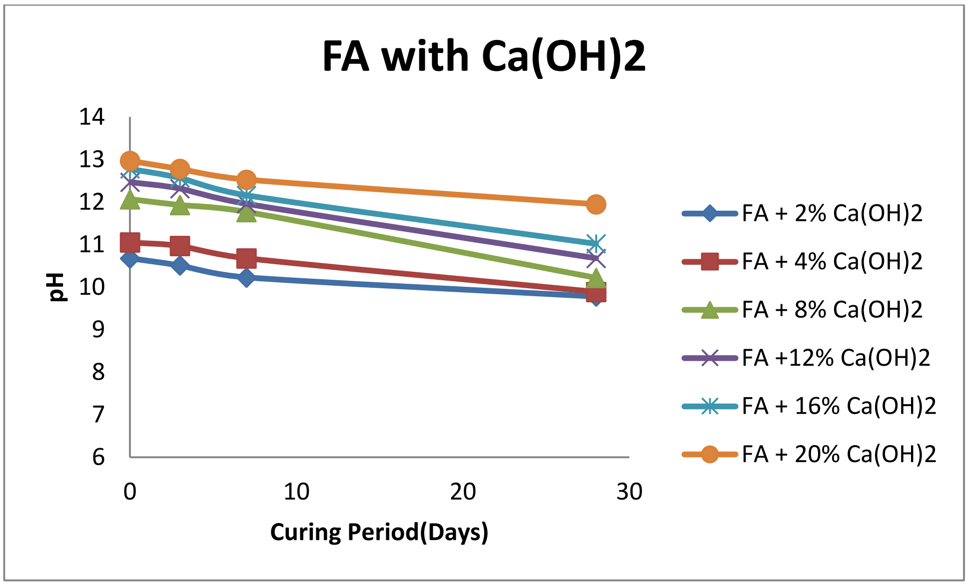



Fig.4.21 (i): Variation of pH with curing period for $Ca(OH)_2$ treated fly ash

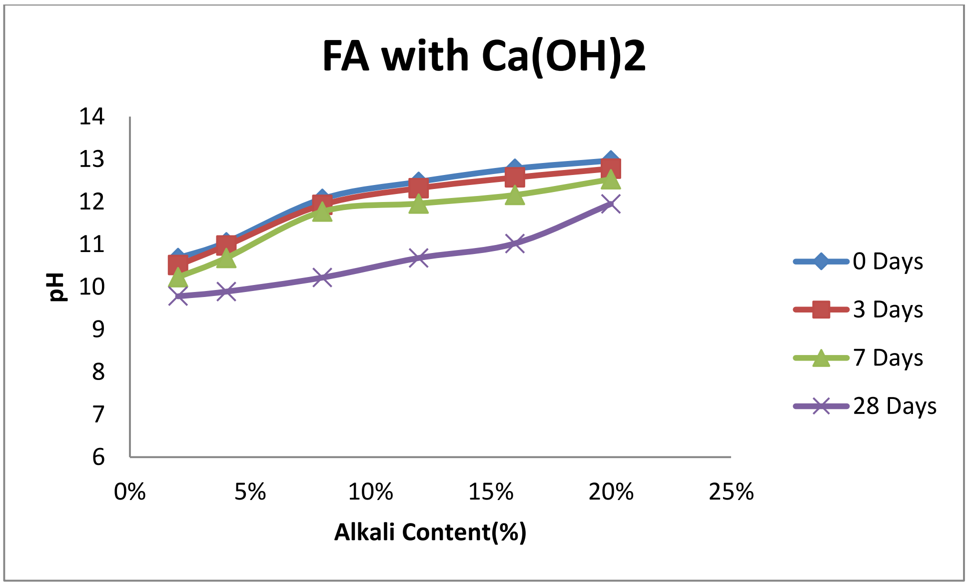



Fig.4.21 (ii): Variation of pH with alkali content for $Ca(OH)_2$ treated fly ash

As the reaction medium is alkaline all the pH value of alkali treated fly ash specimens are around or above 10. From the above figures it can be concluded that pH value of alkali treated fly ash specimens decreases marginally with increase in curing period because when curing period increase more amount of alkali is being used to form the gel, and very obviously pH value of alkali treated fly ash specimens increase as alkali content increase for all three types of alkalis.

## 4.3.5 XRD, SEM and EDX Study

X-ray diffraction (XRD) technique is used for characterization of the compounds formed due to the reaction between alkali and silica ($SiO_2$) and alumina ($Al_2O_3$) present in the fly ash.

Scanning electron microscope (SEM) is used to determine surface topography composition the alkali treated fly ash sample with different alkali type, composition.

Energy dispersive X-ray analyzer (EDX) is used to characterize the mineral present in the alkali treated fly ash sample with different alkali type, composition.

XRD, SEM and EDX study have been conducted for selected samples i.e. fly ash samples treated with 12% NaOH, 16% NaOH, 20% NaOH, 16% KOH, 20% NaOH, 16% $Ca(OH)_2$ and 20% $Ca(OH)_2$ with a curing period of 28 days and shown in the figures 4.22 to 4.28.

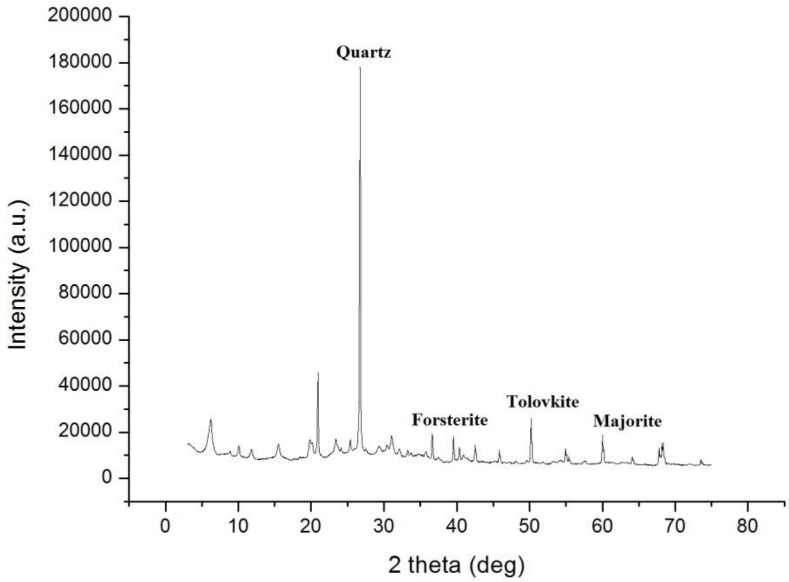
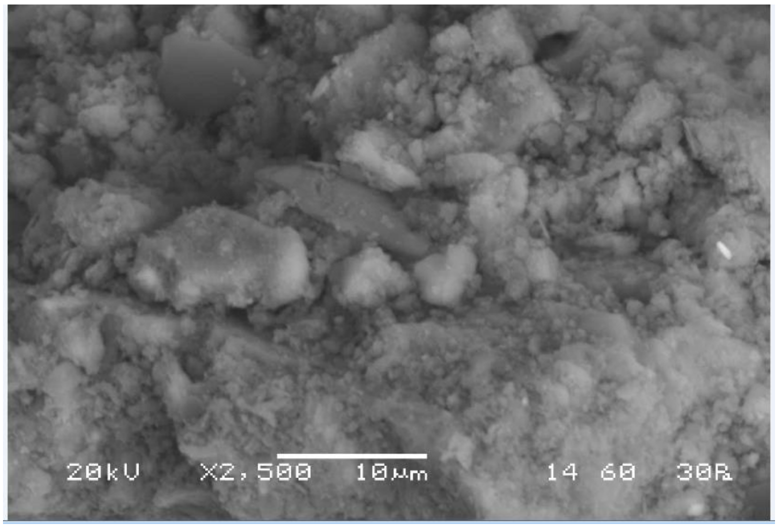



Fig.4.22 (i): XRD of fly ash treated with 12% NaOH cured for 28 days




.4.22 (ii): SEM of fly ash treated with 12% NaOH cured for 28 days

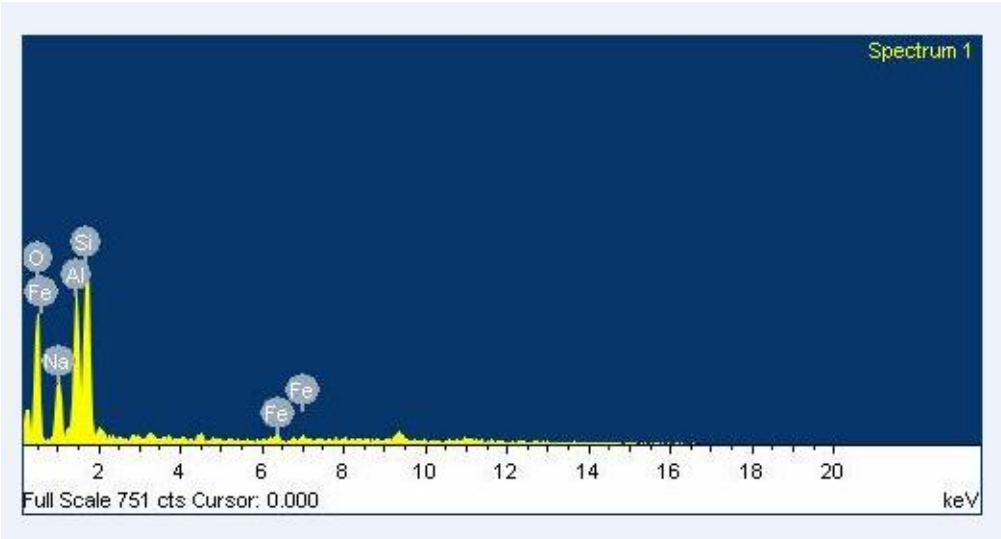



Fig. 4.22 (iii): EDX of fly ash treated with 12% NaOH cured for 28 days

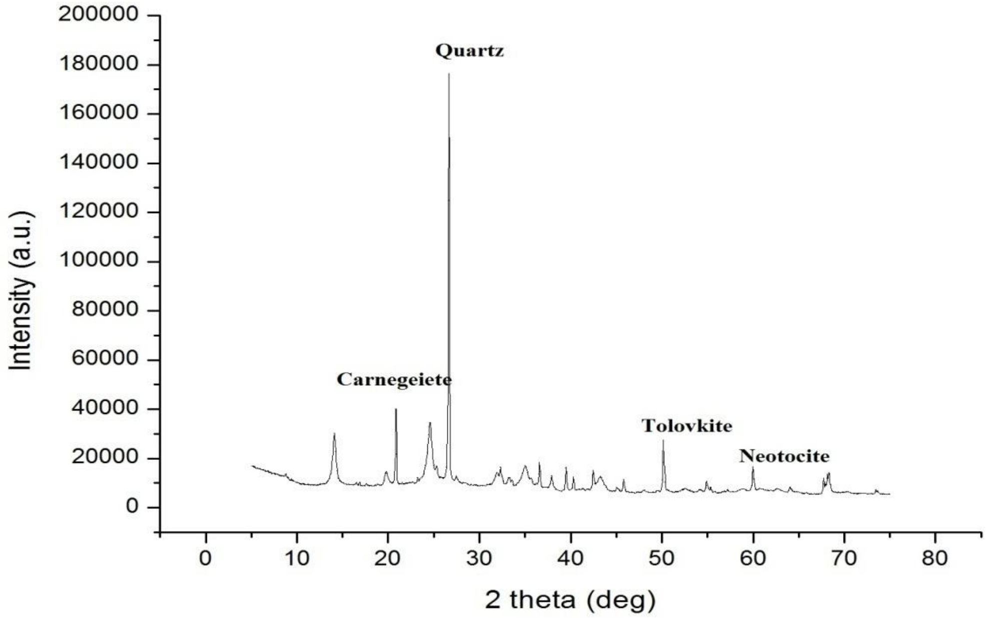



Fig.4.23 (i): XRD of fly ash treated with 16% NaOH cured for 28 days

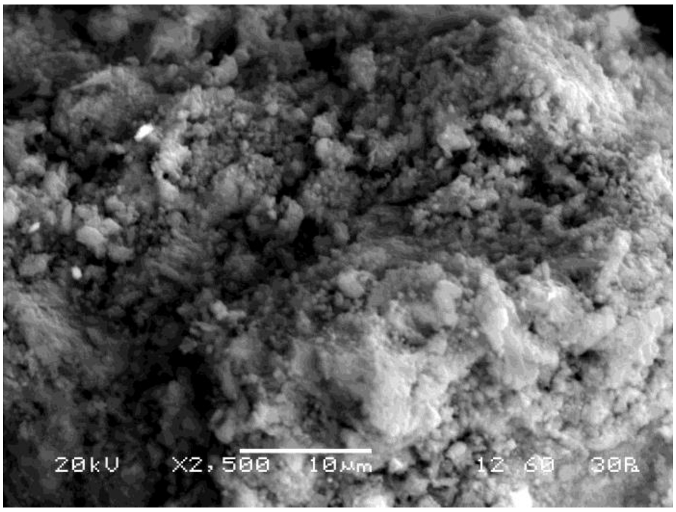



Fig.4.23 (ii): SEM of fly ash treated with 16% NaOH cured for 28 days

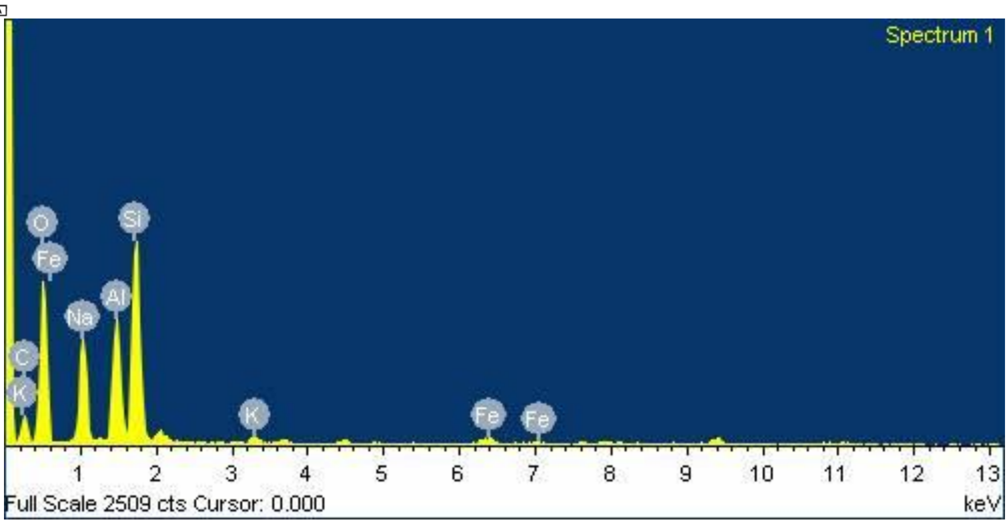



Fig.4.23 (iii): EDX of fly ash treated with 16% NaOH cured for 28 days

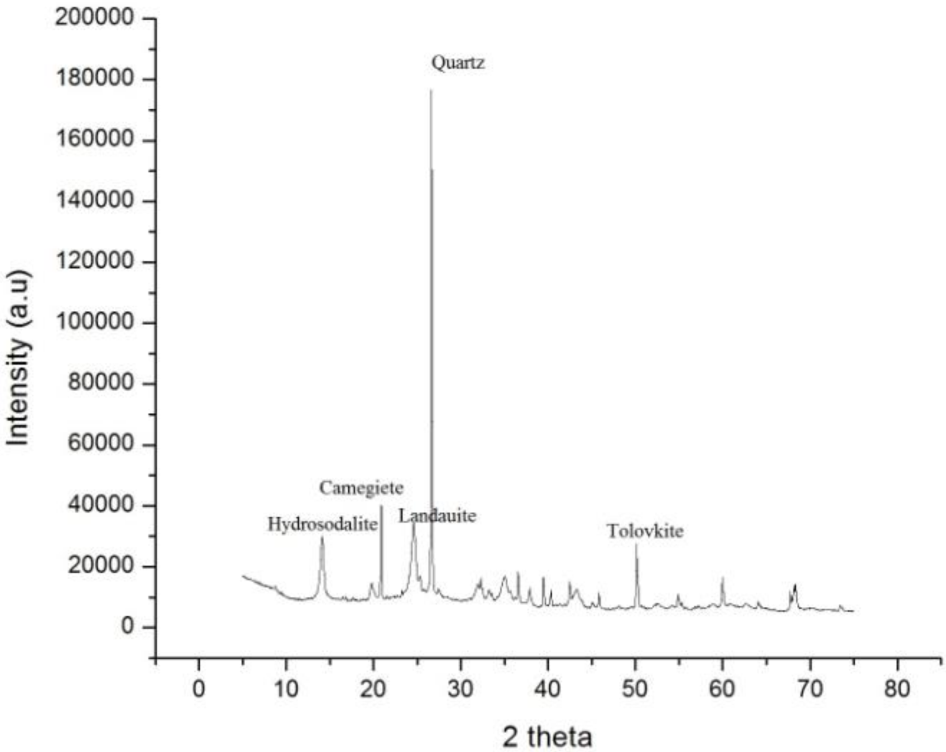



Fig.4.24 (i): XRD of fly ash treated with 20% NaOH cured for 28 days

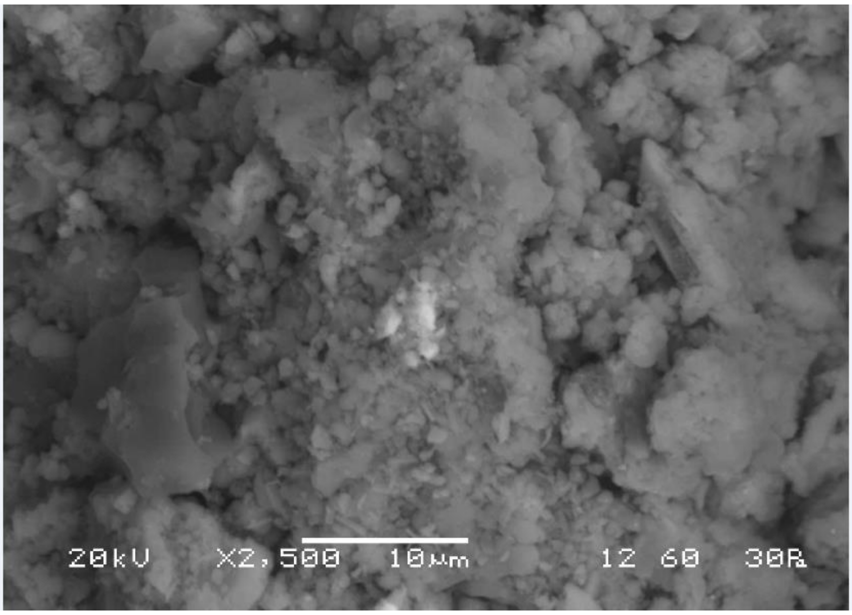



Fig.4.24 (ii): SEM of fly ash treated with 20% NaOH cured for 28 days

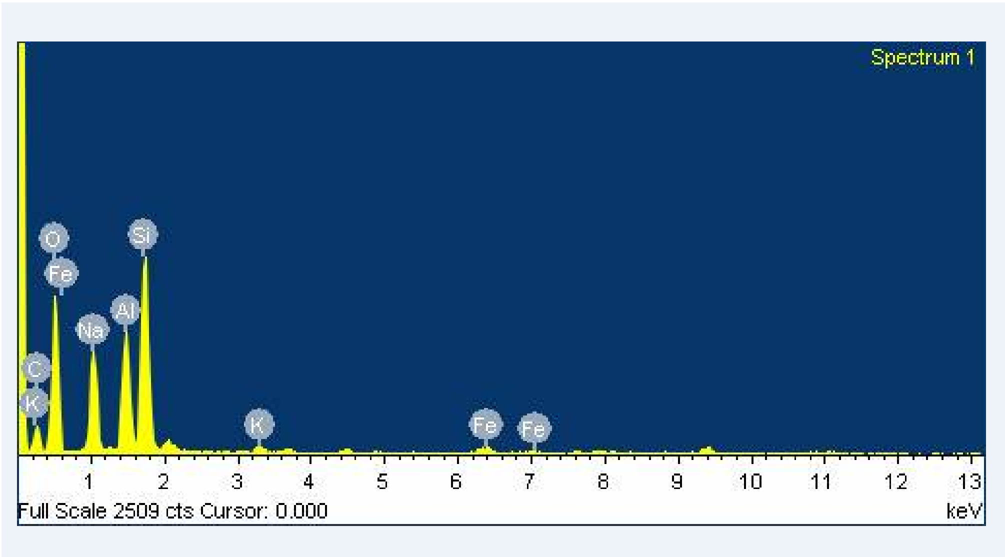



Fig.4.24 (iii): EDX of fly ash treated with 20% NaOH cured for 28 days

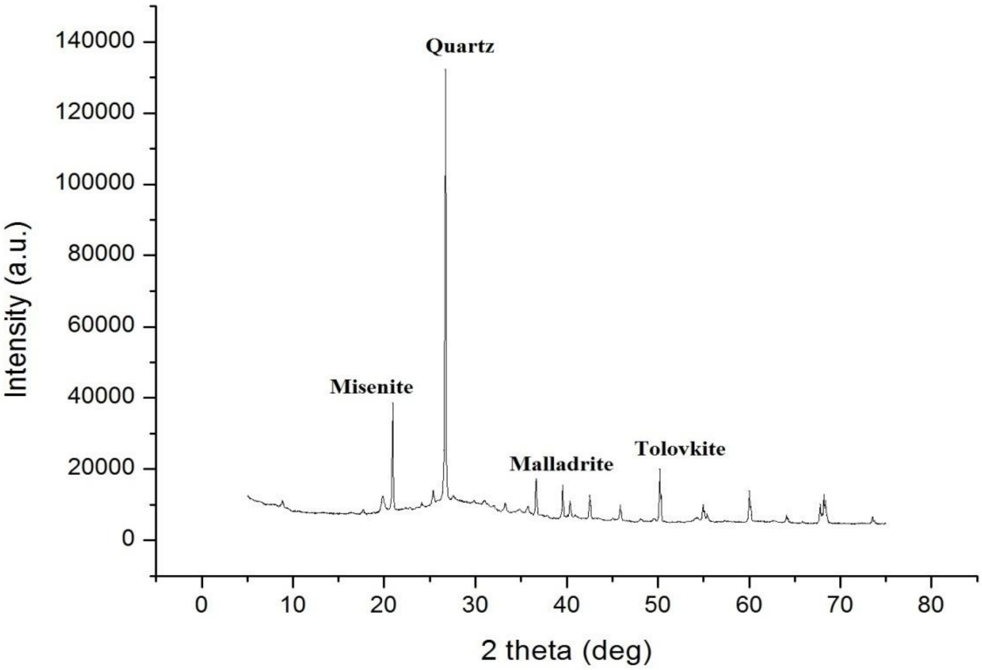



Fig.4.25 (i): XRD of fly ash treated with 16% KOH cured for 28 days

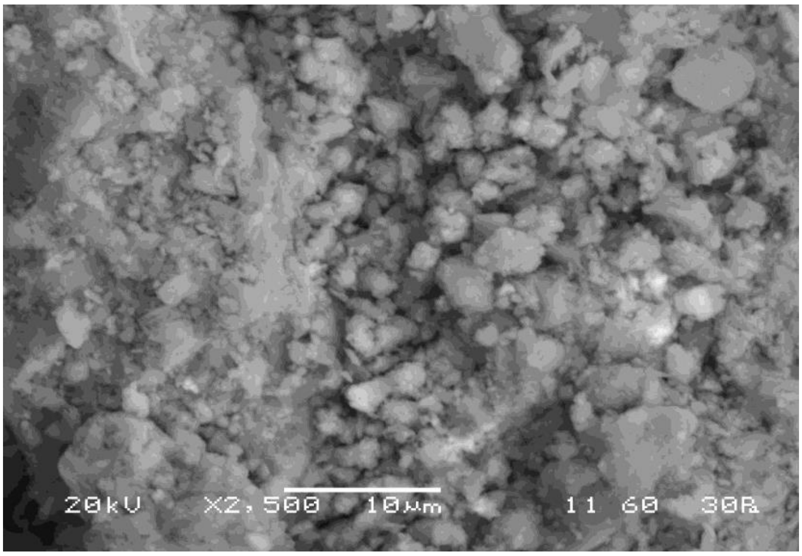



Fig.4.25 (ii): SEM of fly ash treated with 16% KOH cured for 28 days

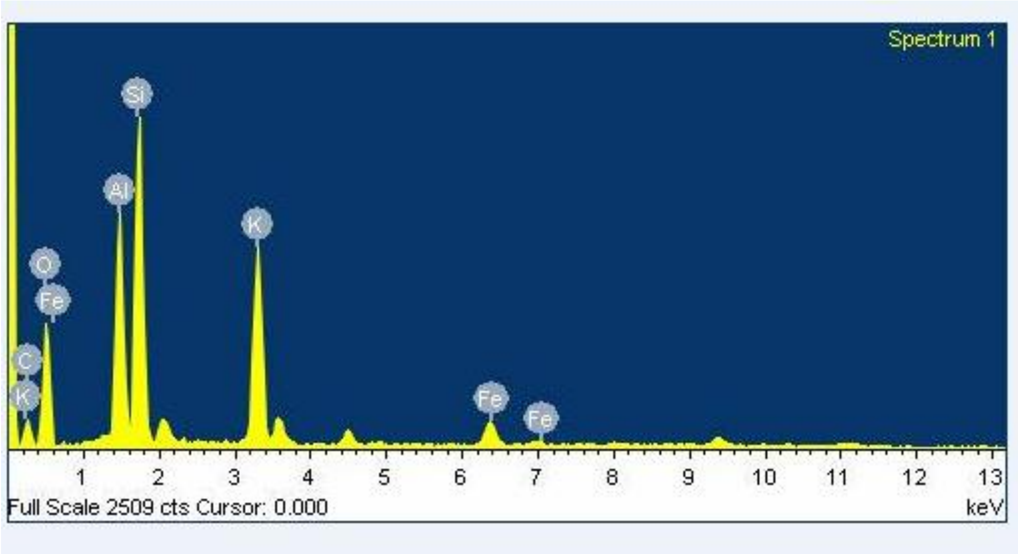



Fig.4.25 (iii): EDX of fly ash treated with 16% KOH cured for 28 days

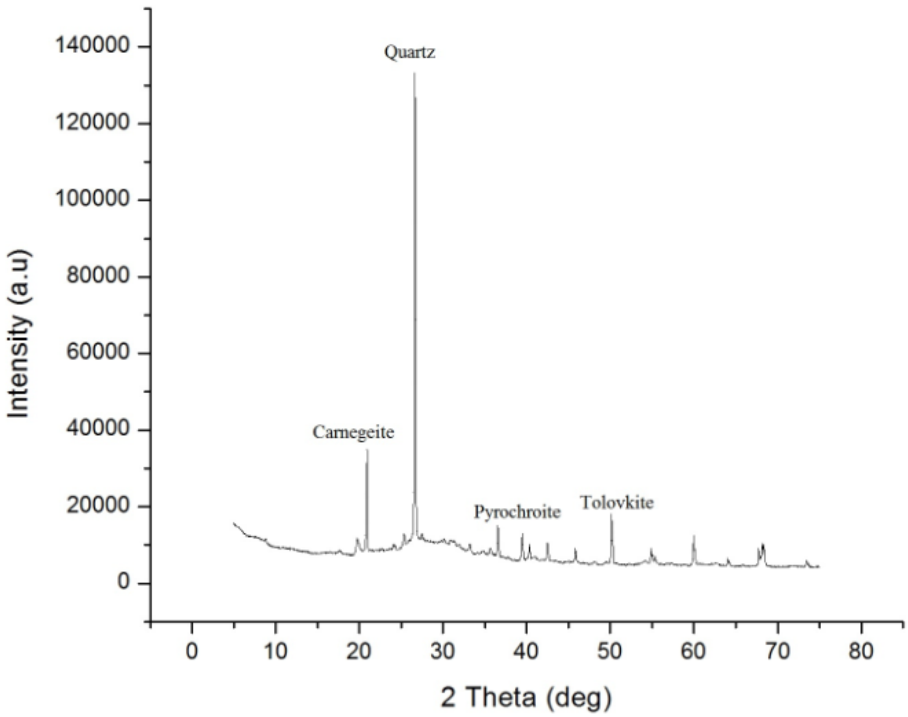



Fig.4.26 (i): XRD of fly ash treated with 20% KOH cured for 28 days

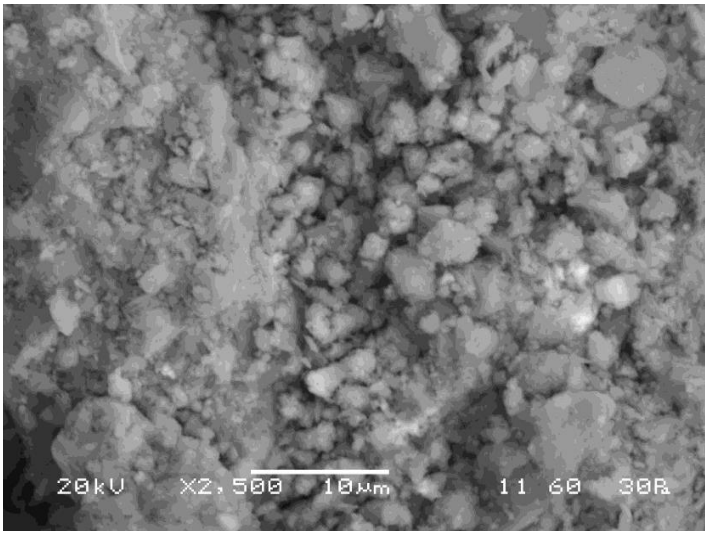



Fig.4.26 (ii): SEM of fly ash treated with 20% KOH cured for 28 days

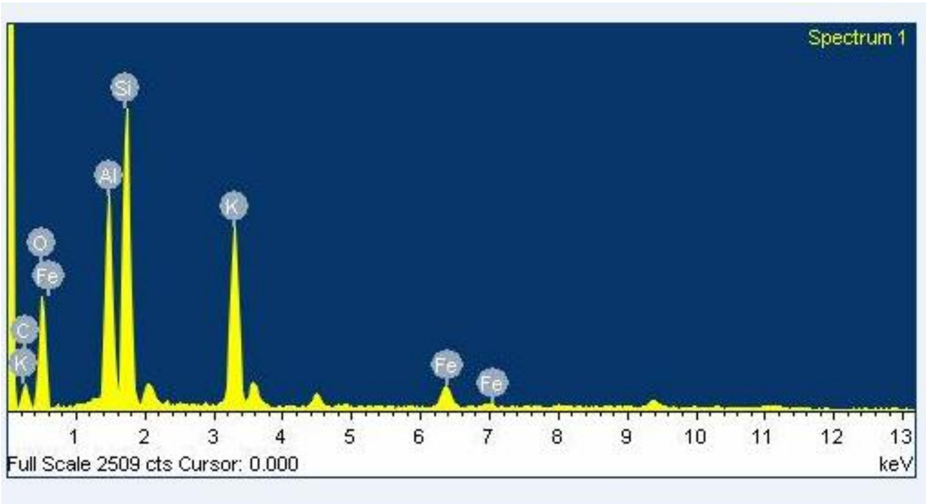



Fig.4.26 (iii): EDX of fly ash treated with 20% KOH cured for 28 days

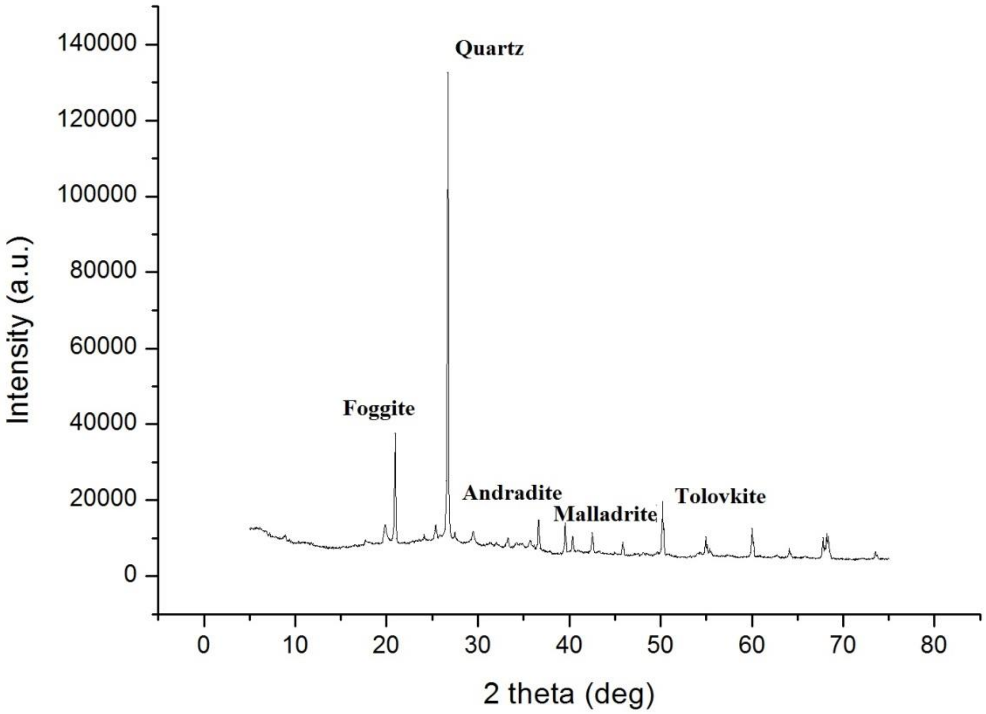



Fig.4.27 (i): XRD of fly ash treated with 16% $Ca(OH)_2$ cured for 28 days

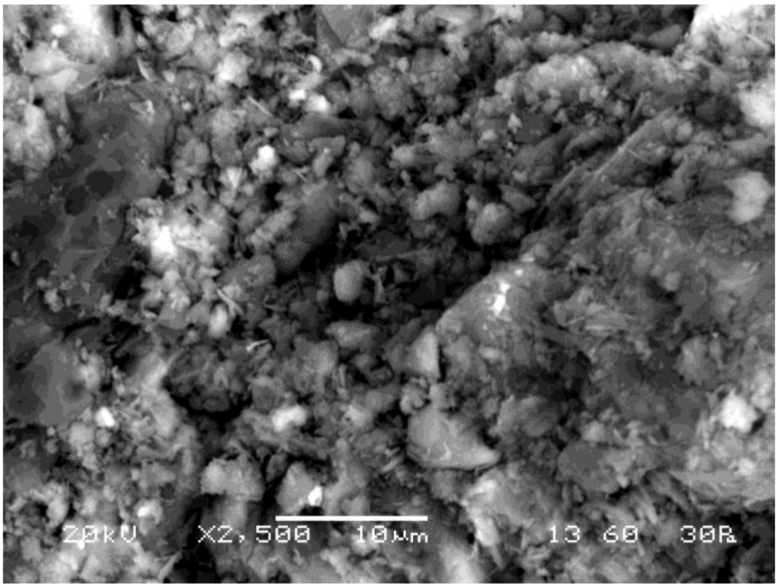



Fig.4.27 (ii): SEM of fly ash treated with 16% $Ca(OH)_2$ cured for 28 days

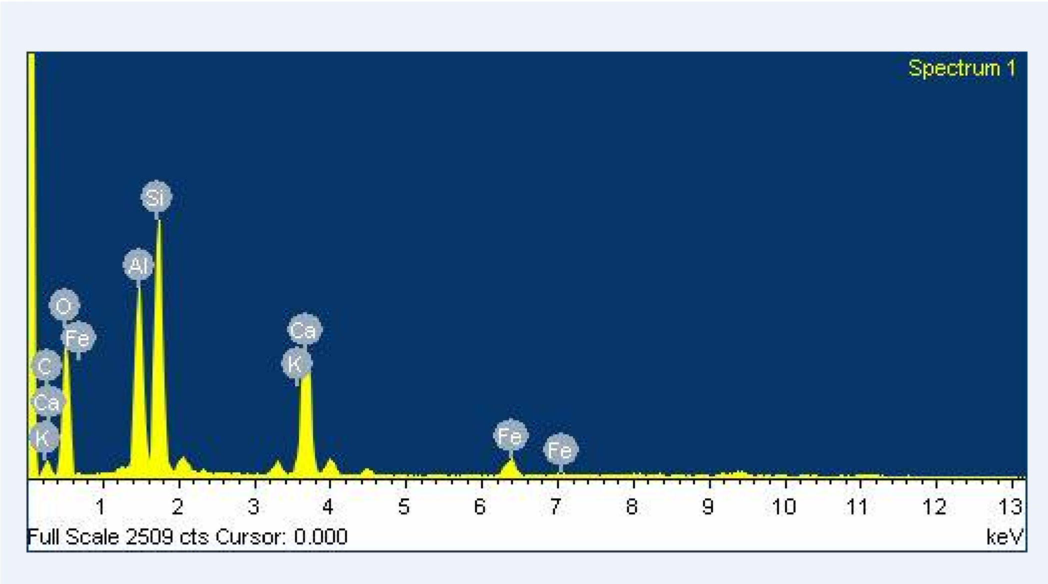



Fig.4.27 (iii): EDX of fly ash treated with 16% $Ca(OH)_2$ cured for 28 days

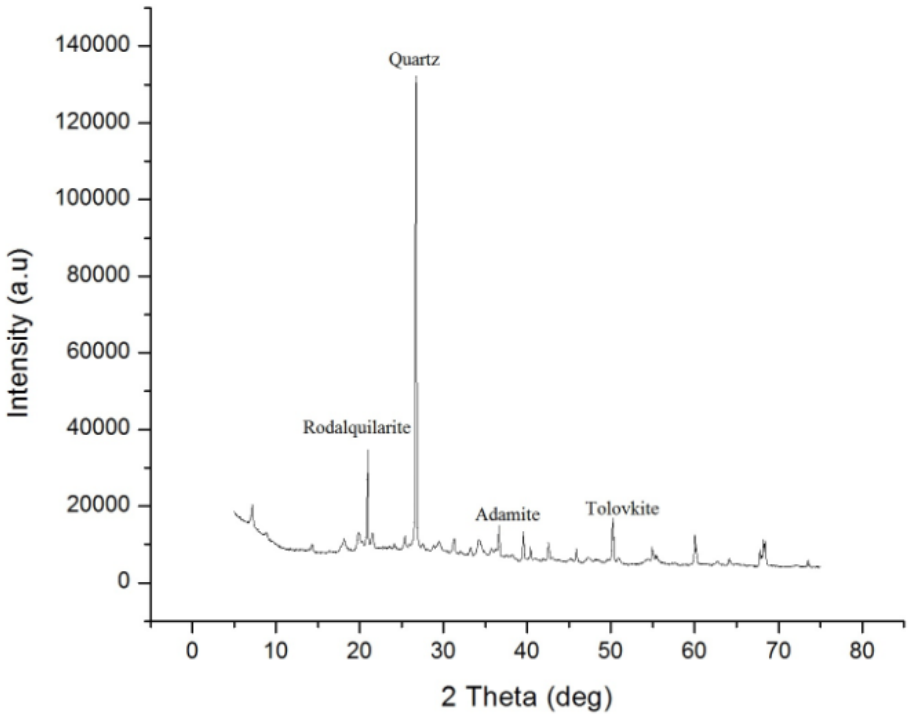



Fig.4.28 (i): XRD of fly ash treated with 20% $Ca(OH)_2$ cured for 28 days

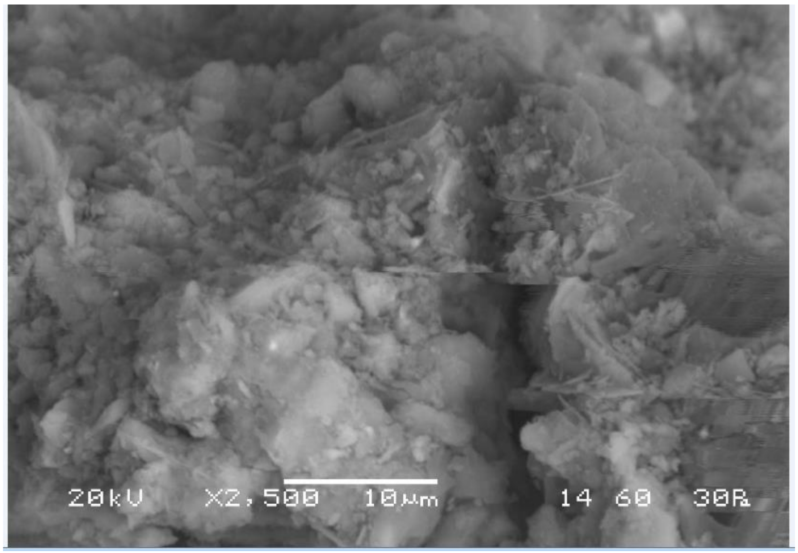



Fig.4.28 (ii) SEM of fly ash treated with 20% $Ca(OH)_2$ cured for 28 days

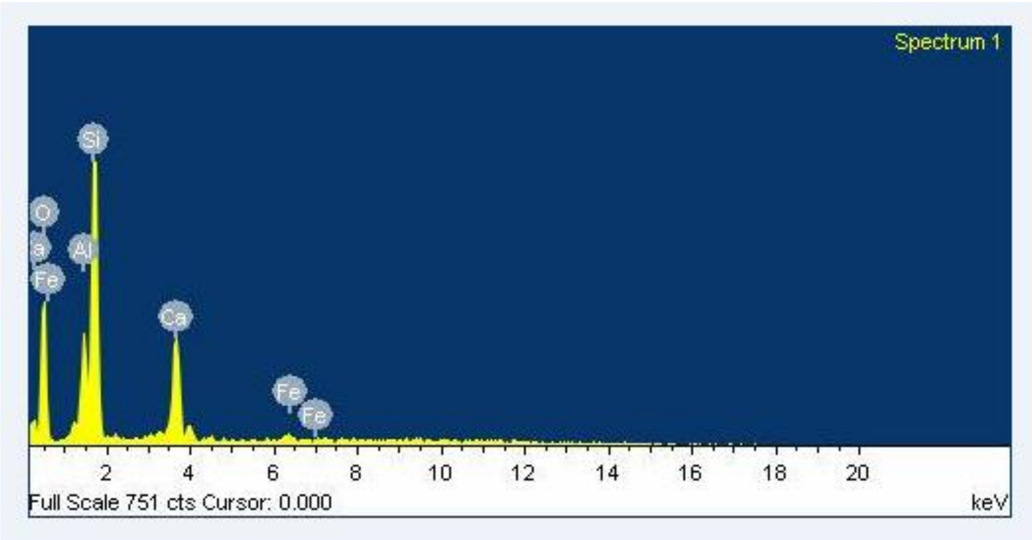



Fig.4.28 (iii) EDX of fly ash treated with 20% $Ca(OH)_2$ cured for 28 days

In all three types of alkali treated fly ash samples quartz is the highest peak as silica is the major compound in the fly ash, the silica which are in crystal state will not react with alkali they remain as quartz.

For 12% NaOH, 16% NaOH and 20%NaOH treated fly ash samples main compound which are found by XRD are Quartz, Carnegeiete, Neotocite, Landauite, Camegiete, Hydrosodalite, Forsterite, Majorite etc. Among them except quartz the general formula of these compounds are $Na_n\{ -(SiO_2)_z -AlO_{2\,-} \}_n$ . So, these are the product of the reaction between NaOH and silica and alumina. From SEM it is observed that the sodium alumino-silicate gel coat the rounded particles of fly ash, a white cotton like substance is there in the picture that is the gel which is responsible for strength. In EDX except Si, Al, Na mineral also found which is obvious.

For 16% KOH and 20% KOH treated fly ash samples main compound which are found by XRD are Quartz, Misenite, Malladrite, Pyrochroite, Tolovkite. Except quartz other compounds are the product of the reaction between KOH and silica and alumina. The general formula of these two compounds are $K_n\{ -(SiO_2)_z -AlO_{2\,-} \}_{n.}$ From SEM it is observed that a white cotton like substance is there that is potassium alumino-silicate gel which coated the rounded particles of fly ash. In EDX except Si and Al K minerals is also found.

For 16% $Ca(OH)_2$and 20% $Ca(OH)_2$ treated fly ash samples main compound which are found by XRD are Quartz, Foggite, Andradite, Malladrite,Rodalquilarite, Adamite. Except quartz other four compounds are the product of the reaction between $Ca(OH)_2$ and silica and alumina. The general formula of these two compounds are $Ca_n\{ -(SiO_2)_z -AlO_{2\,-} \}_{n.}$ From SEM it is observed that a white cotton like substance is there that is calcium alumino-silicate gel which coated the rounded particles of fly ash. In EDX except Si and Al Ca minerals is also found.

# CHAPTER 5: CONCLUSIONS AND FUTURE WORK

## 5.1 Conclusions

Experiments are carried out to investigate strength properties of alkali stabilized fly ash. The effects of alkali type, alkali content, curing period on the strength properties of stabilized fly ash are investigated. Based on the experimental investigations the following main conclusions are arrived at:

- The fly ash consists of most of the particles of fine sand to silt size. The Coefficient of uniformity (Cu) and coefficient of curvature (Cc) for flyash was found to be 5.67 & 1.25 respectively, indicating that it is a uniformly graded material..
- Fly ash treated with alkali (i.e. NaOH and KOH) shows increase in maximum dry density(MDD), decrease in optimum moisture content(OMC), as the theNaOH and KOH solution have some lubricating effect which densifies the fly ash but fly ash treated with Ca(OH)2 doesn't shows much changes in MDD & OMC valus as $Ca(OH)_2$ is added in powder form and water is added separately and water doesn't possess that much of lubricancy as NaOH and KOH solution.
- A general increase in UCS values are observed for fly ash stabilized with NaOH , KOH, $Ca(OH)_2$with curing period and alkali content. This is because of alkali alumino-silicate gel is formed due to the reaction between alkali and silica and alumina present in fly ash which filled up the pores in the sample and solidify the material, resulting in increment in strength.
- Though some surprising behaviour also been observed i.e. for NaOH stabilized fly ash highest UCS value is obtained at 12% NaOH content 70 days curing after that at 16% and 20% NaOH content the UCS value decreases; for $Ca(OH)_2$ stabilized fly ash highest UCS value is obtained at 20% $Ca(OH)_2$ content 28 days curing after that at 70 days curing theUCS value decreases. This type of characteristics is shown may be because after achieve a optimum strength the excess gel which is formed try to exert on the sample resulting in decrement in strength.
- The values co-efficient of permeability (k) obtained from falling head permeability test are constantly decreasing with increase in alkali content and curing period, because the alkali alumino-silicate gel filled all the pores in the stabilized fly ash

- sample and close the flow path of water resulting in decrement in co-efficient of permeability (k).
- As the reaction medium is alkaline initially the pH value of alkali treated fly ash sample is very high, then it decreases marginally with the curing period because during the reaction process alkali is being used so pH decreases.
- From XRD analysis it is found that the reaction compounds are alkali almino-silicate gel with a general formula $M_n\{ -(SiO_2)_z -AlO_{2\,-} \}_n$ which is responsible for strength; where M represents the alkali group i.e. Na, K, Ca.
- In SEM it is observed that white gel like substance coated the fly ash particle and EDX study shown that except Si and Al
- For NaOH stabilized fly ash after 70 days of curing at $27^0$C temperature we can achieve 24803 Kpa at 12% NaOH content strength which is equivalent to concrete strength. For KOH stabilized fly ash after 70 days of curing at $27^0$C temperature we can achieve 12614 Kpa strength at 20% KOH content. For $Ca(OH)_2$ stabilized fly ash after 28 days of curing at $27^0$C temperature we can achieve 16504 Kpa strength at 20% $Ca(OH)_2$ content. Even at low alkali content we can achieve desired strength of alkali stabilized fly ash to be used as a replacement of conventional earth like for 4% NaOH content we achieve 1672 Kpa and 2968 Kpa strength at 28 days and 70 days curing.
- After 28 days of curing at $27^0$C temperature we can achieve value of co-efficient of permeability(k) as 1.5x10^-8, 3.8x10^-8, 2.53x10^-8 corresponding to 20% NaOH, 20% KOH and 20% $Ca(OH)_2$ stabilized fly ash, which is much below than conventional earth.
- At the end after observing the results it can be concluded that at lower percent alkali stabilized fly ash can be used as a replacement of good earth material on which construction is possible and at higher percent alkali stabilized fly ash can be used as a replacement of concrete also with which construction can be done, though still further investigation is necessary on this topic.
- Alkali stabilized fly ash can be used as a alternate construction material or geo-material in civil engineering construction.

## 5.2 Future Work

- Behavior of alkali stabilized fly ash under the action of repeated loading can be studied.
- Durability test of alkali stabilized fly ash sample can be performed.
- Compressibility and Consolidation characteristics of alkali stabilized fly ash can be performed.
- Effect of alkali on the leachate which comes from the fly ash can be studied.
- Liquefaction test of alkali stabilized fly ash sample can be performed.
- Except this investigation can be performed with different types of industrial waste material i.e. red mud, pond ash, blast furnace slag etc.

## PUBLICATIONS FROM THIS WORK

- **Chowdhury S.**,Tiwari A., Singh S.P.,Pani A.**,**"**Alkali Activated Fly Ash: A New Generation Geo-Material**", 5th Young Indian Geotechnical Engineers Conference 2015, pp 105-106.

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